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FERRITES, MAGNETIC, H. F.

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AS AD NO.

Report No. 57

Contract No. 6

Signal Corps Contract: DA-36-039
SC-89222

Dept. of Army Task Number IGO-24401-A-112-02-03
(DA99-15-006-02)

Sixth Quarterly Report

1 Sep 1963 to 30 November 1963

U.S. ARMY ELECTRONICS RESEARCH AND DEVELOPMENT LABORATORY
Fort Monmouth, New Jersey

INDIANA GENERAL CORPORATION

ELECTRONICS DIVISION
RESEARCH DEPARTMENT

KEASBEY, NEW JERSEY — Telephone Valley 6-5100,



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Report No. 57 Contract No. 6
Signal Corps Contract DA-36-039
 SC-89222

1 September 1963 to 30 November 1963

REPORTED BY: Dr. Eberhard Schwabe, Physicist - Supervisor
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Daniel Sullivan, Ceramic Engineer
Charles O'Neill, Chemist
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ABSTRACTS

PART I

MANGANESE-ZINC FERRITES

SECTION A

Effect of Vibration on μ_0 , Q, and Temperature Coefficient of Permeability

Certain production and experimental materials were subjected to a vibration test. μ_0 , Q and temperature curve measurements were made before and after 10 and 100 minutes vibration. No significant effect on these properties was observed that could be ascribed solely to vibration.

Time Dependence of Temperature Coefficient of Permeability

Certain production and experimental materials were measured over a 24 hour period at each of three different temperatures. Although changes in permeability were noted after each 24 hour period, no evidence was obtained to indicate that the slope of the temperature curve had changed.

SECTION B

The annealing study initially presented in Report No. 56 has been continued during this past quarter but will not be discussed until the next report.

PART II

NICKEL-ZINC FERRITES

SECTION A

Effect of Vibration on μ_0 , Q, and Temperature Coefficient of Permeability

Certain production and experimental materials were subjected to a vibration test. μ_0 , Q and temperature curve measurements were made before and after 10 and 100 minutes vibration. No significant effect on these properties was observed that could be ascribed solely to vibration.

Time Dependence of Temperature Coefficient of Permeability

Certain production and experimental materials were measured over a period of 24 hours at each of three different temperatures.

ABSTRACTS
(continued)

Although changes in permeability were noted after each 24 hour period no evidence was obtained to indicate that the slope of the temperature curve had changed.

SECTION B

Investigation of the Effects of Particle and Grain Size on μ_0Q , Temperature Coefficient and Disaccommodation of Ni-Zn Ferrites in the Frequency Range, 0.1 - 500 mc/s

The study on Ni-Zn ferrites presented in Report No. 56, Section B of Part II, was continued. Temperature coefficient data was obtained for material MF 9002 and no improvement over MF 9001 was noted. Disaccommodation data on MF 9002 was also obtained and here significant improvement was noted over MF 9001. Chemical analysis of MF 9002 was completed and the amount of iron milled into this material was found to be less than the amount of iron milled into MF 9001. Chemical analysis of MF 9001-A, 64 hour milling and 128 hour milling, was also completed and the formulas obtained from the analysis agree quite closely with the formula for MF 9001, 64 hour milling. This explains in part the similarity of properties found previously. Material MF 9003 was made and several measurements of μ_0 and Q were taken. This data will be completed and presented in the next report.

PART I - Mn-Zn FERRITES

SECTION A

EFFECT OF VIBRATION ON μ_o , Q AND TEMPERATURE COEFFICIENT OF PERMEABILITY

During the past quarter, at the request of USAELRDL, certain production and experimental materials were subjected to a limited scope vibration test. It was hoped that some insight would be gained regarding the stability of these materials after being subjected to a stress of this sort. Ordinarily, specifications call for vibration tests at various frequencies and for varying periods of time. Our test, however, was limited to one frequency. The equipment used was a standard laboratory vibration mill which has a frequency of vibration of approximately 45 cycles per second. The samples to be vibrated were attached with masking tape to the outside of the milling jars. Two time periods were used for the vibration tests, 10 minutes and 100 minutes.

The samples selected for the test were the production material TC-5 and the experimental materials MF-8433-1, MF-8433-2, MF-8644-1 and MF-8644-4. These samples were first measured for μ_o and Q at 100, 200 and 400 kc/s and then measured at 100 kc/s to determine their temperature coefficient. After these measurements the samples were vibrated for 10 minutes and remeasured for μ_o , Q and temperature coefficient; then they were vibrated for 100 minutes and remeasured.

Graph 705 shows the results of this vibration test on the $\mu_o Q$ product at various frequencies. The initial, 10 and 100 minute curves for each material are practically superimposed. Very little change is observed. In addition to the graph on the $\mu_o Q$ product, the values of μ_o and Q are plotted separately as a function of frequency before and after vibration and are shown in graph 706.

The temperature curves of these materials, shown in graphs 707 through 711, reveal little differences. The character of the curves for each material remains about the same, with perhaps a slight displacement observable. This displacement, however, can hardly be ascribed solely to vibration, since, simply remeasuring the materials without first vibrating them could yield differences of the same magnitude as observed in these graphs. In general, this last comment holds for all results obtained on these vibration tests.

TIME DEPENDENCE OF TEMPERATURE COEFFICIENT OF PERMEABILITY

At the request of USAELRDL the permeability of certain materials was measured over a 24 hour period at each of three different temperatures. Three Mn-Zn type materials were included in this test, the production material TC-5, and the experimental materials MF-8433-2 and MF-8644-4. These materials were initially demagnetized and then placed in the temperature chamber at 25°C. They were allowed 20 minutes to adjust to the temperature and then measured. After this initial measurement the samples were held at 25°C for 24 hours and remeasured. The temperature of the chamber was then raised to 50°C and, after allowing 20 minutes for the samples to adjust to the temperature, a measurement was taken. The samples were held for another 24 hours at this temperature, and then another measurement was taken. The chamber was then lowered to -40°C, and the same procedure as above was repeated.

Table 296 shows a tabulation of the results obtained. The data clearly shows changes in the permeability values after the 24 hour period. This, of course, was expected, since these materials are known to disaccommodate. However, it is questionable that the temperature coefficient itself has been affected, since there is no evidence from this test that the slope of the temperature curve has changed.

SECTION B

INVESTIGATION OF THE EFFECTS OF PARTICLE AND GRAIN SIZE
ON μ_{coQ} , TEMPERATURE COEFFICIENT AND DISACCOMMODATION OF
Mn-Zn FERRITES IN THE FREQUENCY RANGE. 1 kc/s TO 1 mc/s

The annealing study presented in Report No. 56 has been continued during this past quarter but will not be discussed until the next report.

PART II - Ni-Zn FERRITES

SECTION A

EFFECT OF VIBRATION ON μ_o , Q AND TEMPERATURE COEFFICIENT OF PERMEABILITY

The vibration test discussed earlier in this report on page 8 also concerned several Ni-Zn or "Q" type materials. These materials were Q-1, Q-2 and TC-4 (all production materials), and MF-9001-128, MF-9002-128 and MF-9003-128 (experimental, non cobalt containing materials; the -128 indicating milling time in hours). Measurements were first taken of μ_o and Q at the frequencies .8, 1.6, 3.2, 6.4 and 12.8 mc/s. Then temperature coefficient measurements were made at the frequency of 1 mc/s. Then the procedure of vibration and measurement described on page 8 was followed.

The $\mu_o Q$ product versus frequency for each vibration period is plotted in graph 712. Again, the curves show very little change. In graph 713, μ_o and Q are plotted separately as a function of frequency before and after vibration.

In graphs 714 through 718, the temperature curves of Q-1, TC-4, MF-9001-128, MF-9002-128 and MF-9003-128 are shown with vibration period as a parameter. The character of these curves for each material is essentially the same, and only a slight displacement is observable. The changes noted during these vibration tests are not sufficient to suggest that vibration has an influencing effect on the electrical and magnetic properties of the materials tested.

TIME DEPENDENCE OF TEMPERATURE COEFFICIENT OF PERMEABILITY

The test described on page 9 of this report also included some "Q" type materials: Q-1, TC-4, MF-9001-128 and MF-9002-128. The test included two each of the Q-1 and TC-4 samples. All samples were demagnetized prior to the test except one Q-1 and one TC-4 sample. The results are compiled in table 296.

There are definite changes in the permeability values after 24 hours. Again, however, it is questionable that the temperature coefficient itself has been affected, since there is no evidence from this test that the slope of the temperature curve has been changed. This sort of evidence would have to come from a temperature curve measurement taken on each of several days.

SECTION B

INVESTIGATION OF THE EFFECTS OF PARTICLE AND GRAIN SIZE ON $\mu_o Q$, TEMPERATURE COEFFICIENT AND DISACCOMMODATION OF Ni-Zn FERRITES IN THE FREQUENCY RANGE, 0.1 TO 500 MC/S

Reference Report No. 56 (Part II, Section B, page 20). Material MF-9002 has been further investigated and measurements have been taken on disaccommodation, density, grain size, the effects of annealing, and the temperature variation of permeability. This data together with previously reported magnetic data are compiled in table 297.

Graphs 719 through 726 show the change of permeability with temperature. Each graph is for a particular milling time of the material and contains several curves corresponding to different firing temperatures. The first four graphs concern materials calcined at 1205°C, and the next four concern the materials calcined at 1260°C. The values of $(\Delta\mu/\mu^2\Delta T)$ for the "optimum curves" materials (see Report No. 56, page 23) are tabulated below.

<u>MF-9002</u>	<u>1205°C Calcine</u>			<u>1260°C Calcine</u>	
	<u>Milling</u> <u>Time</u>	<u>Firing</u> <u>Temp.</u>	<u>$\frac{\Delta\mu}{\mu^2\Delta T}$</u>	<u>Firing</u> <u>Temp.</u>	<u>$\frac{\Delta\mu}{\mu^2\Delta T}$</u>
	16	1300°C	9.8	1300°C	13.6
	32	1250°C	10.7	1250°C	10.3
	64	1250°C	17.5	1250°C	16.4
	128	1215°C	18.1	1200°C	17.4

The two calcining temperatures disclose essentially equivalent results. In comparison with material MF-9001 (see Report No. 56, page 20) the $(\Delta\mu/\mu^2\Delta T)$ values are higher except for the 128 hour milled material, which has values approximately the same.

Graphs 727 through 730 show disaccommodation curves for MF-9002. As found previously with MF-9001, the disaccommodation effect increases with increasing milling time. Generally, disaccommodation is lower for the MF-9002 materials than for the MF-9001 materials. Particularly, the best MF-9001 material showed a disaccommodation value ($\Delta\mu/\mu^2$ per decimal cycle) of 107.5 ppm, while the best MF-9002 material shows a value of 72.5 ppm. The general decrease noted suggests that by increasing the Ni/Zn ratio even more, still lower disaccommodation values may be obtained. A material with a

higher Ni/Zn ratio has been prepared, MF-9003, but disaccommodation values will not be available until the next report.

In Report No. 56 the chemical analysis of MF-9001 was discussed. The analysis showed that a considerable amount of Fe_2O_3 was incorporated into the calcined ferrite material during the milling process. An increase of up to 6 weight % was observed, and this was with the 128 hour milled material. MF-9002 has also been chemically analyzed and an increase in the iron content with extended milling has again been observed. The results of this analysis are presented in table 298. The amount of milled in iron is significantly less than observed before for MF-9001, viz., by almost 35%. No reason for this is offered except to say that perhaps the amount of iron milled into the calcined material is a random phenomenon which results from the fact that all the factors affecting the milling operation are not strictly controlled, e.g., temperature, viscosity of mix, steady uninterrupted milling, etc.

The molar Ni/Zn ratios for the various millings remained fairly constant and averaged .921 for the 1205°C calcine and .969 for the 1260°C calcine. The higher value of this ratio for the 1260°C calcine can be explained by the fact that at this higher temperature a slightly greater loss of zinc was incurred.

In Report No. 56, page 22, the material MF-9001-A was discussed. This material was prepared to duplicate the formula of MF-9001 except that the iron content was adjusted so that milling for 64 hours would bring it to ~ 50 mol%. This experiment was tried in order to find out what influence this excess iron oxide had on the properties of MF-9001 so that a proper evaluation of the effect of fine grain size could be made.

A chemical analysis of MF-9001-A has been completed, and the results are compiled in table 299. The analysis showed an iron content which is quite close to that obtained for MF-9001, i.e., the 64 and 128 hour milled material of MF-9001-A showed an iron content close to that for the 64 hour milled material of MF-9001, e.g.,

Milling Time	Fe_2O_3 (wt%)	
	MF-9001	MF-9001-A
64	70.21	69.72
128	73.06	71.20

This explains why the results found for both millings of MF-9001-A were so close to those found for MF-9001, 64 hour milling. An examination of the weights of material used for the starting material of MF-9001-A revealed that it was quite close to MF-9001. This means that the deficiency in iron thought to be introduced actually was not. This leaves the question still open concerning

the excess iron, and it is expected that this will be resolved during the next quarter.

During the past quarter material MF-9003 was prepared, and some μ_o and Q measurements were taken. These measurements are not yet complete, but they will be completed during the next quarter. Also, temperature coefficient and disaccommodation measurements will be taken.

A summary of best results in the 2-12 mc/s range is listed below.

	Q-1		MF-9001 1205°C Calcine 128 hour milling		MF-9002 1205°C Calcine 128 hour milling	
	2 mc/s	12 mc/s	2 mc/s	12 mc/s	2 mc/s	12 mc/s
μ_o	169	210	195	250	180	220
Q	190	20	149	8.2	158	12.7
$\mu_o Q$	32,000	4,200	29,000	2,050	28,500	2,800
UUF (mc/s)	6.1		5.1		6.8	
$\mu_o Q \cdot$ UUF	91,500		81,000		83,000	
T_C $\frac{\Delta \mu}{\mu \Delta T}$ (ppm/°C)	710 ⁱ		3400 ⁱⁱ		3300 ⁱⁱ	
$\frac{\Delta \mu}{\mu^2 \Delta T}$	4.8 ⁱ		17.4 ⁱⁱ		18.1 ⁱⁱ	
$\frac{\Delta \mu_o}{\mu_o}$	1.7		2.0		1.3	
$\frac{\Delta \mu}{\mu^2}$ DA/cycle (ppm)	140.5		107.5		72.5	

1. -30°C to 90°C ($\Delta \mu / \mu \Delta T = 9000$ from -60°C to -30°C)
 ii. -60°C to 90°C

PART III

RESEARCH PLANNED FOR NEXT QUARTER

Part I Mn-Zn Ferrites

- A. No future work will be done in this section.
- B. The annealing study presented in Report No. 56, Section B of Part II will be continued.

Part II Ni-Zn Ferrites

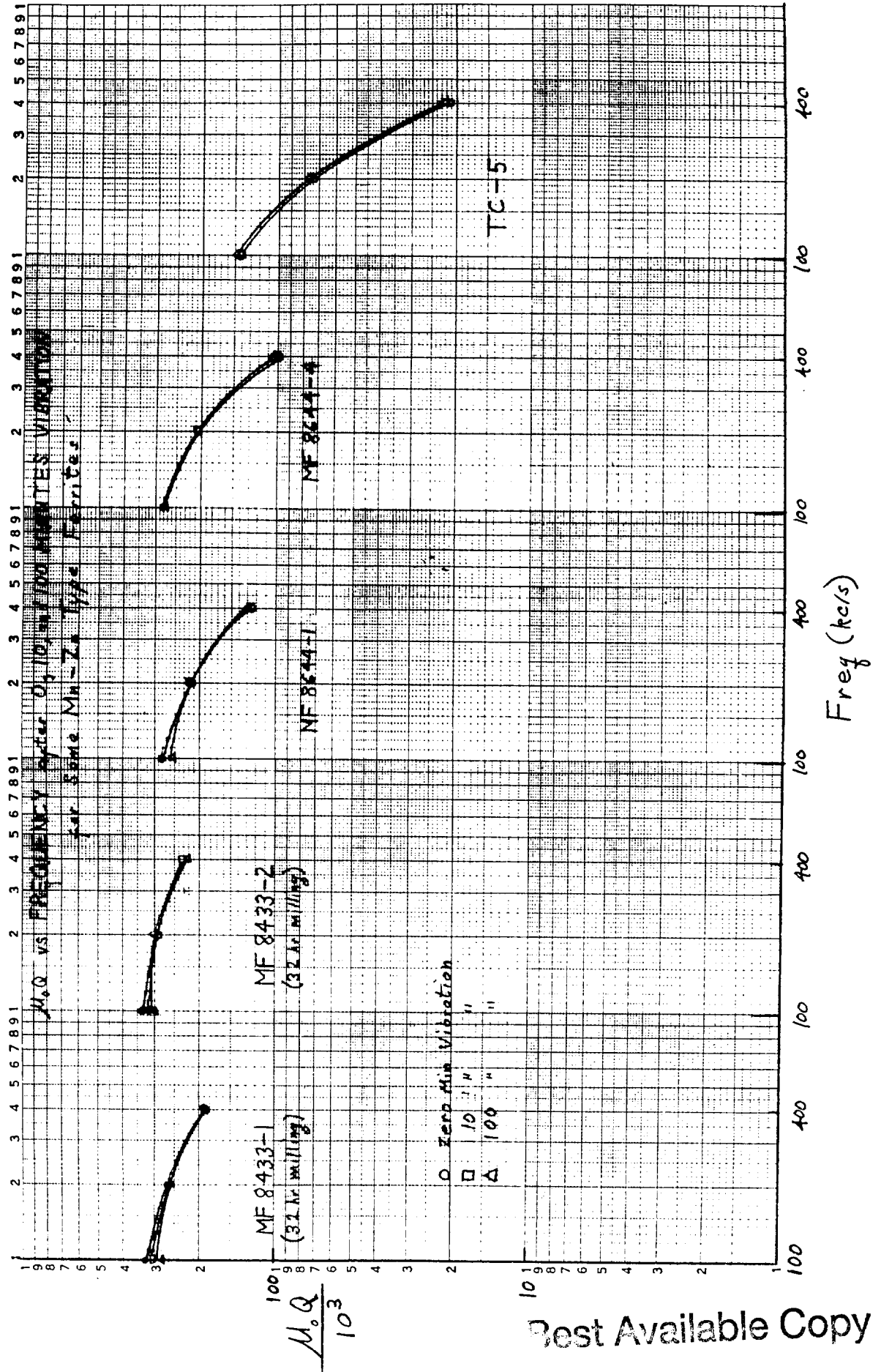
- A. Variations of MF-9002 will be made using nickel compounds of differing reactivity. Short milling periods and production kilns for firing will be utilized. It is desired to see if these preparative techniques can yield a material better than MF-9002, which requires long milling to obtain optimum properties.
- B. Material MF-9003 will be further studied in regards to temperature coefficient and disaccommodation. Firing time and annealing time experiments will be conducted.

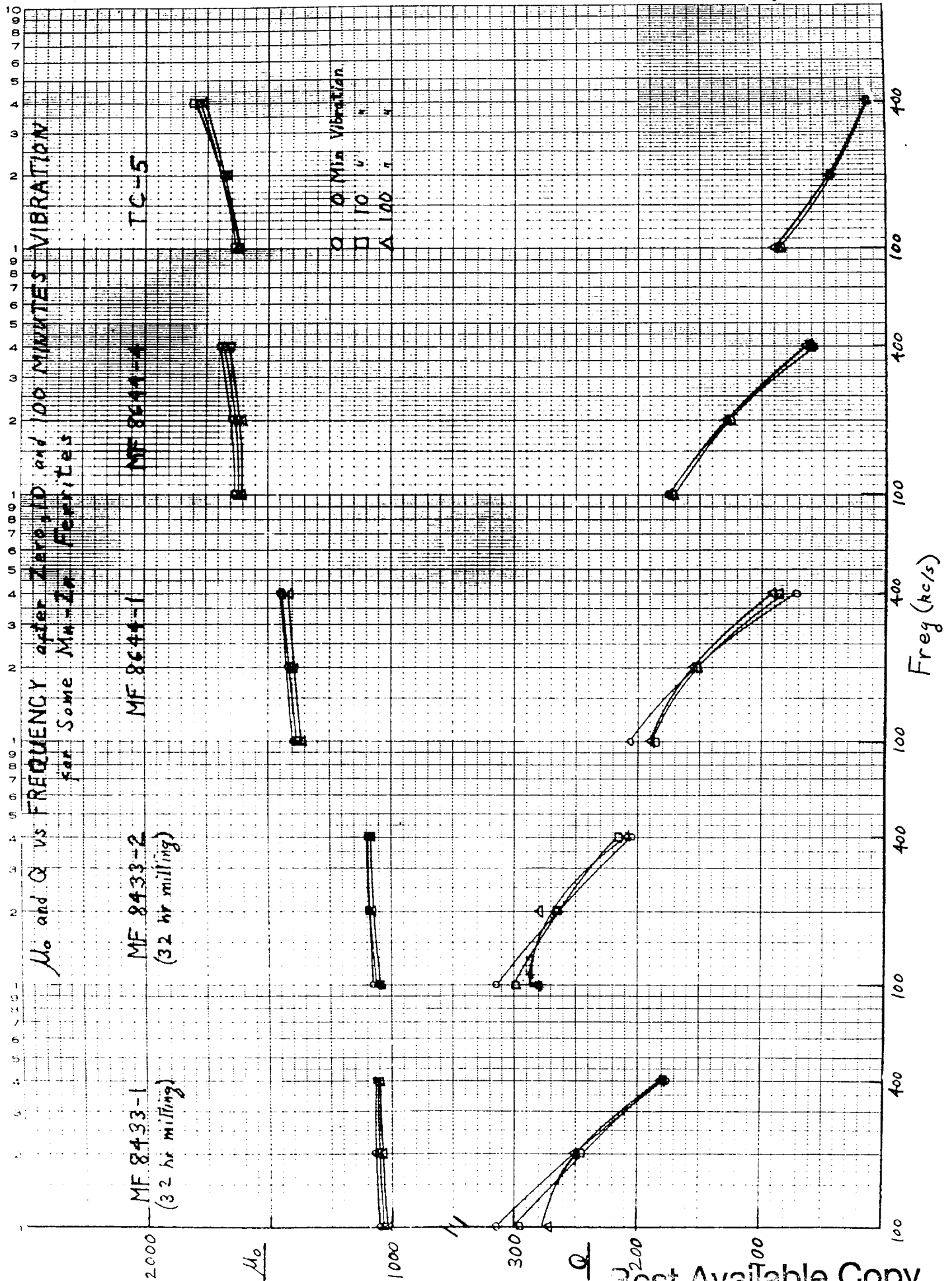
PART IV

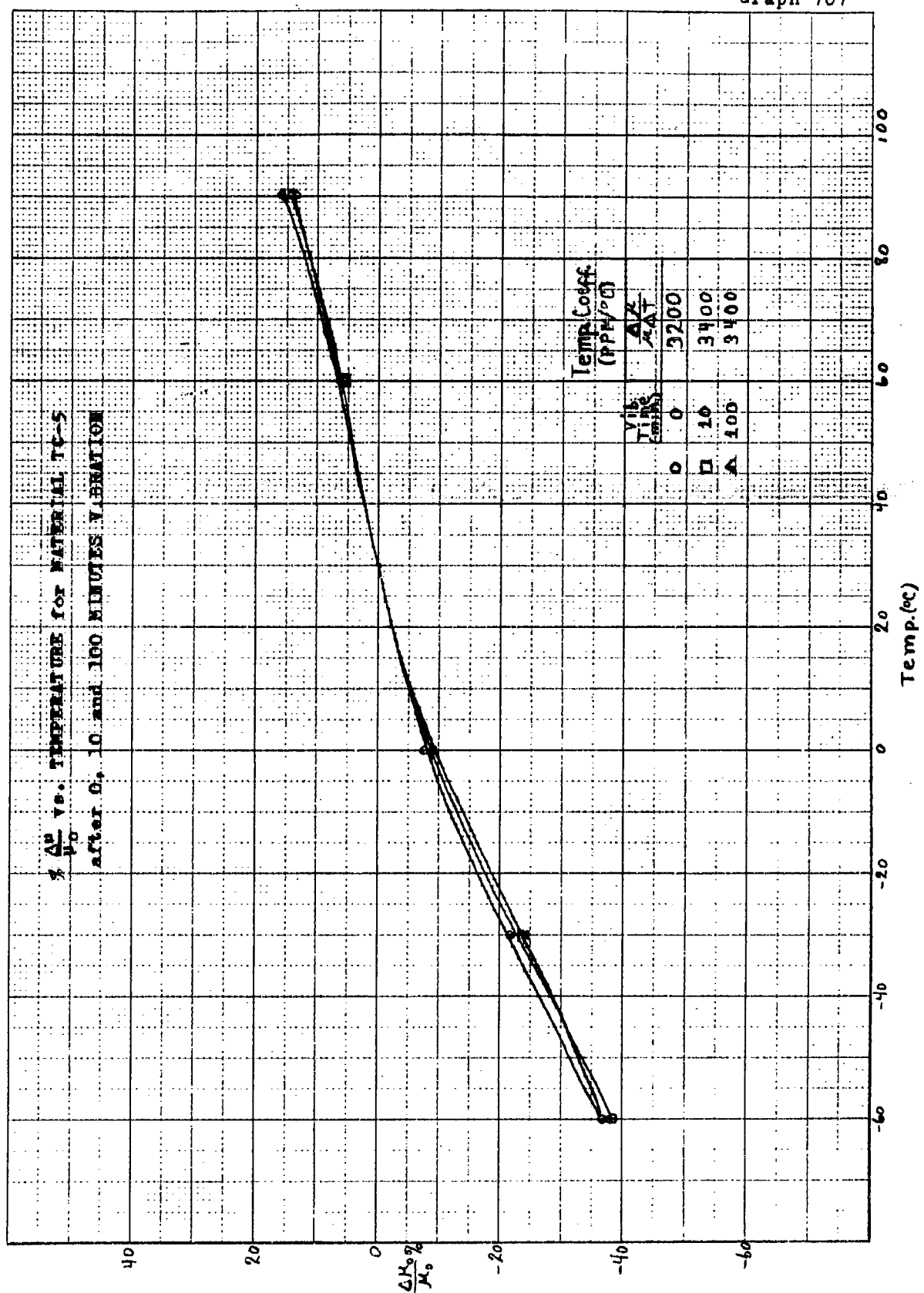
MANHOURS SPENT ON CONTRACT FOR THE PERIOD
1 SEPTEMBER 1963 TO 30 NOVEMBER 1963

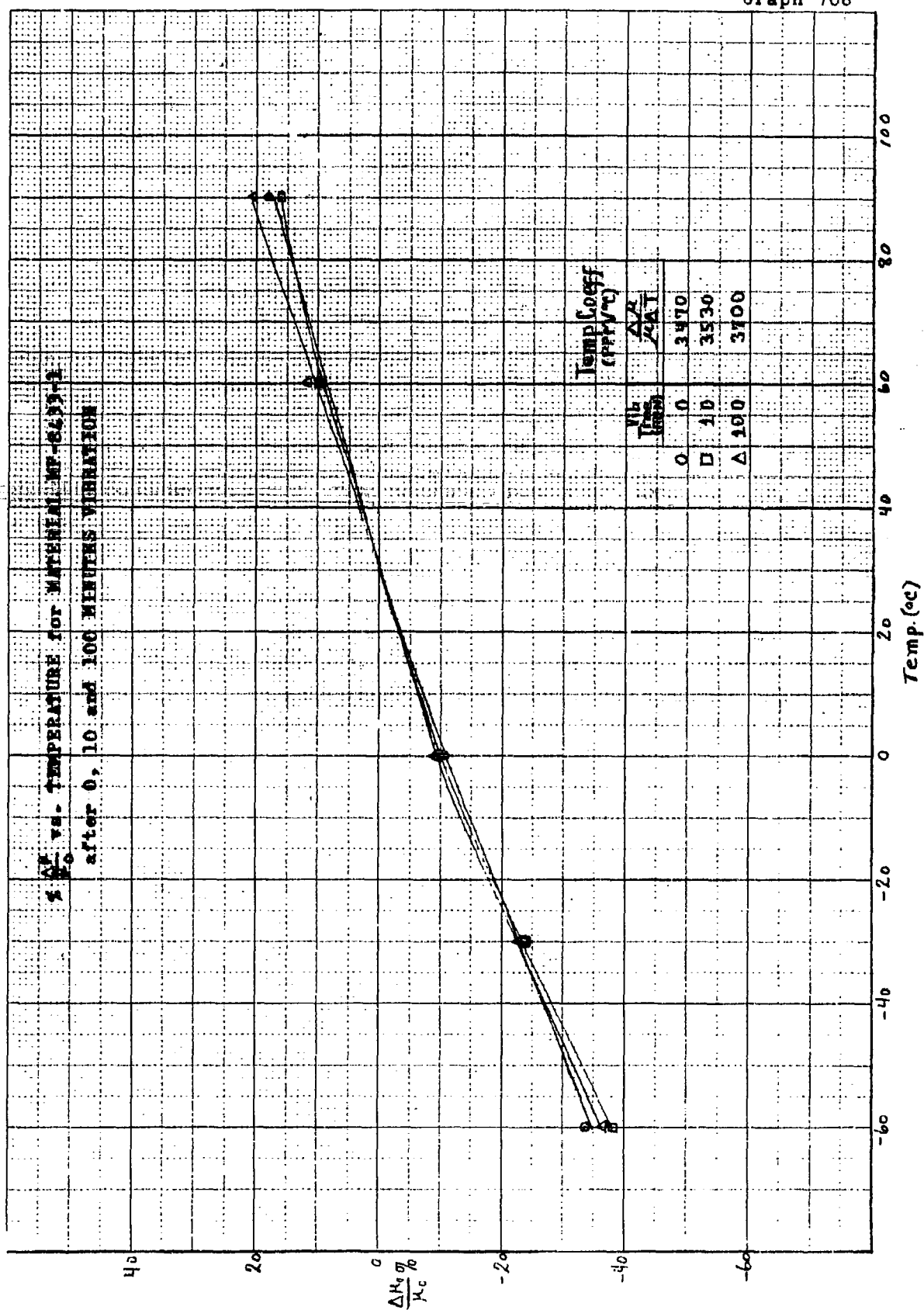
<u>NAME</u>	<u>TITLE</u>	<u>HOURS</u>
E. Schwabe	Physicist - Supervisor	158
K. Wetzel	Chemist	440
C. O'Neill	Chemist	460
D. Sullivan	Ceramic Engineer	477-1/2
S. Golian	Ceramic Engineer	78
K. Sivak	Chemist, Junior	243
C. Cooper	Technician	1-1/2
J. Holden	Technician	480
E. Hozeny	Technician	238
D. Kinsley	Technician	269-1/2
E. Kovacs	Technician	301-1/2
C. Lotz	Technician	165
E. Saatkowski	Technician	199
M. Zudonyi	Technician	20
S. Rubarski	Laboratory Assistant	2

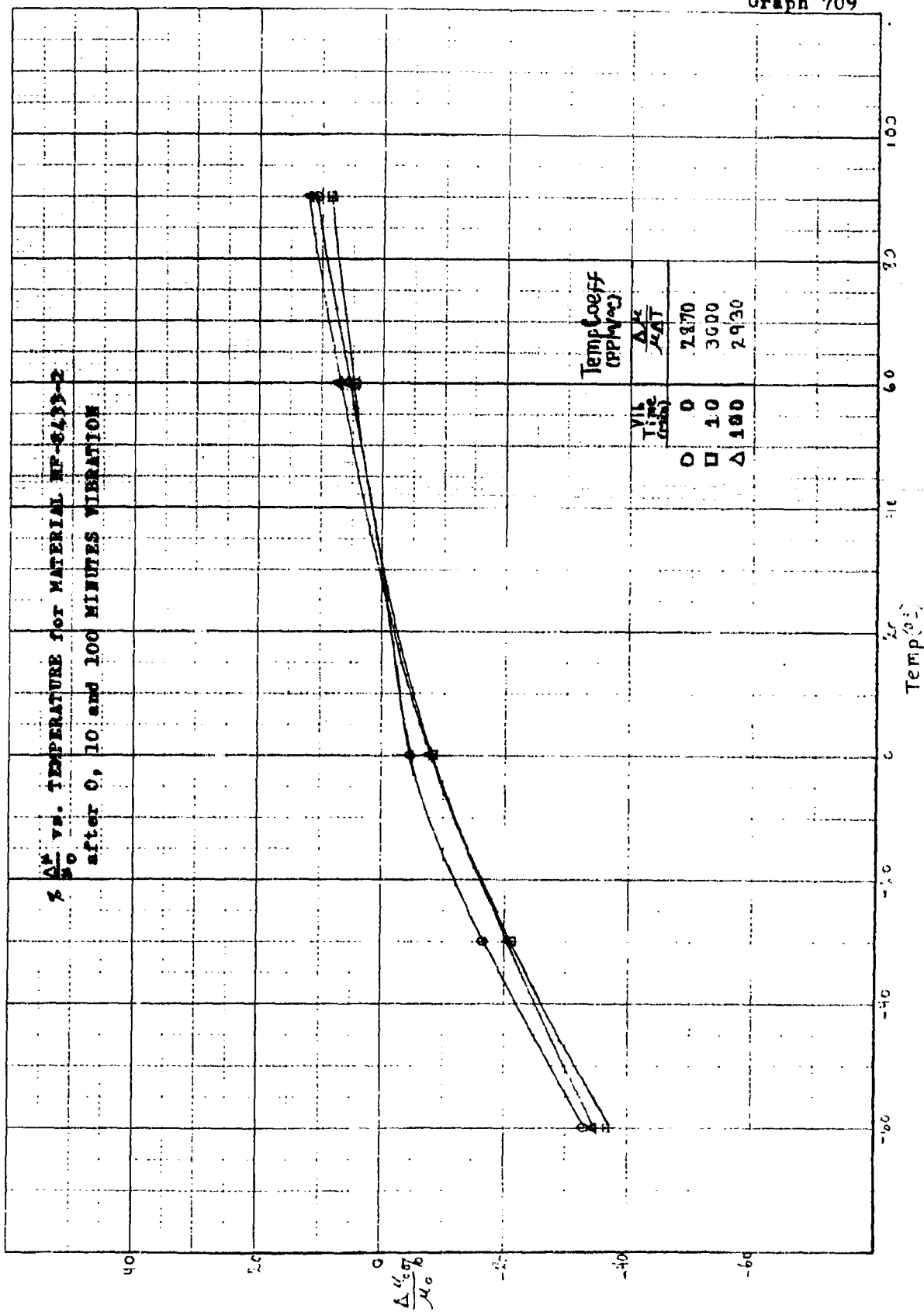
Graph 705



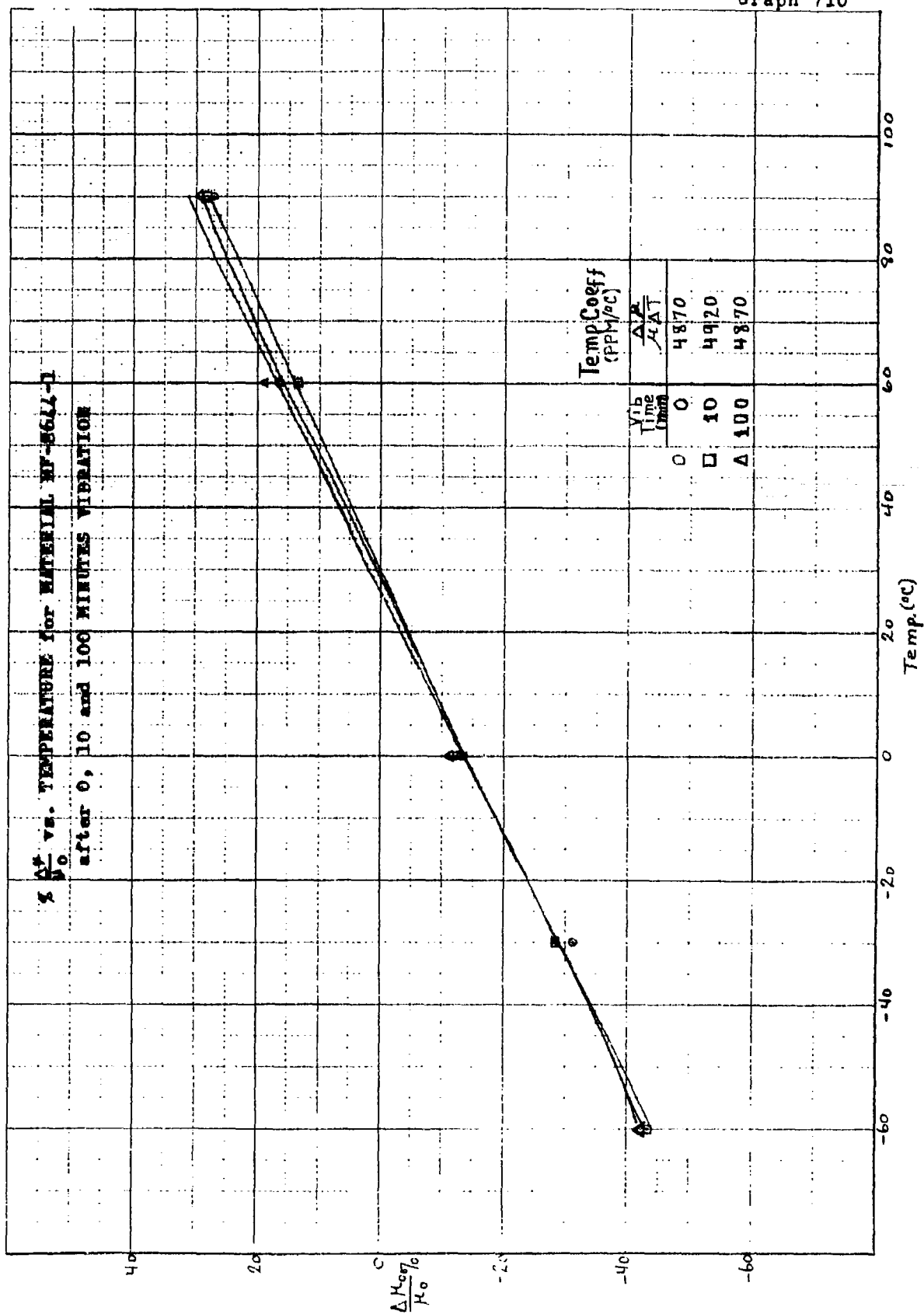




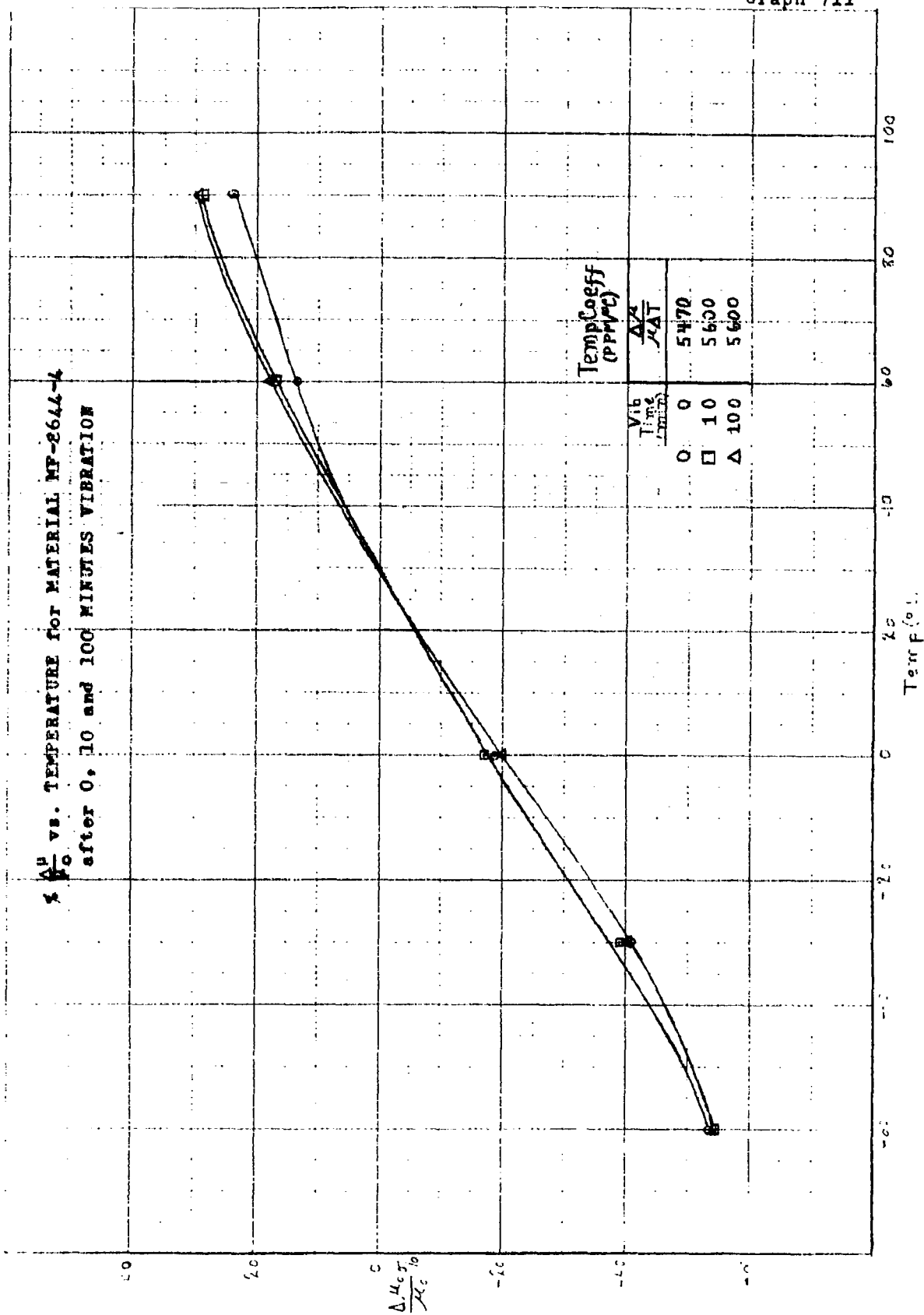




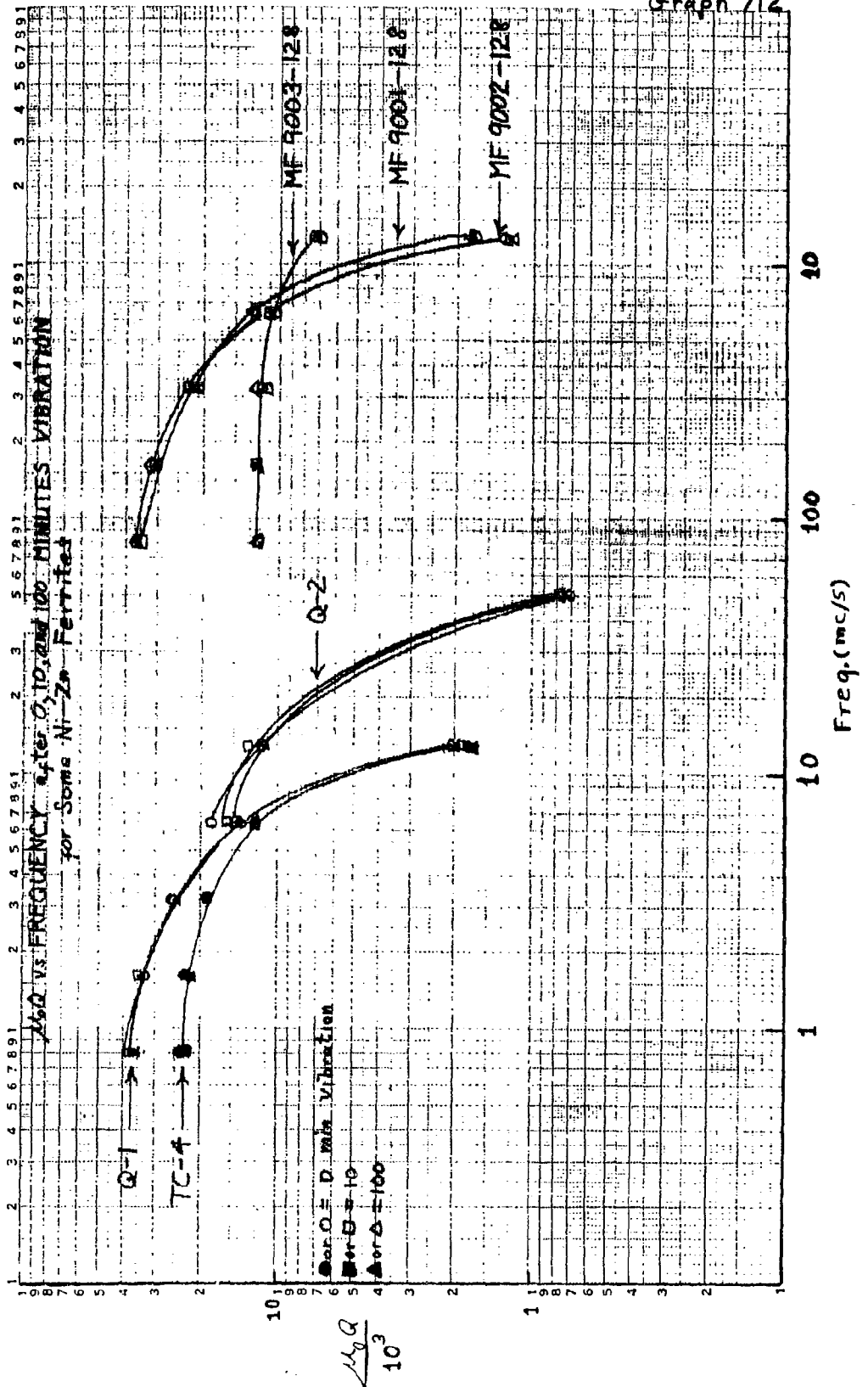
Graph 710



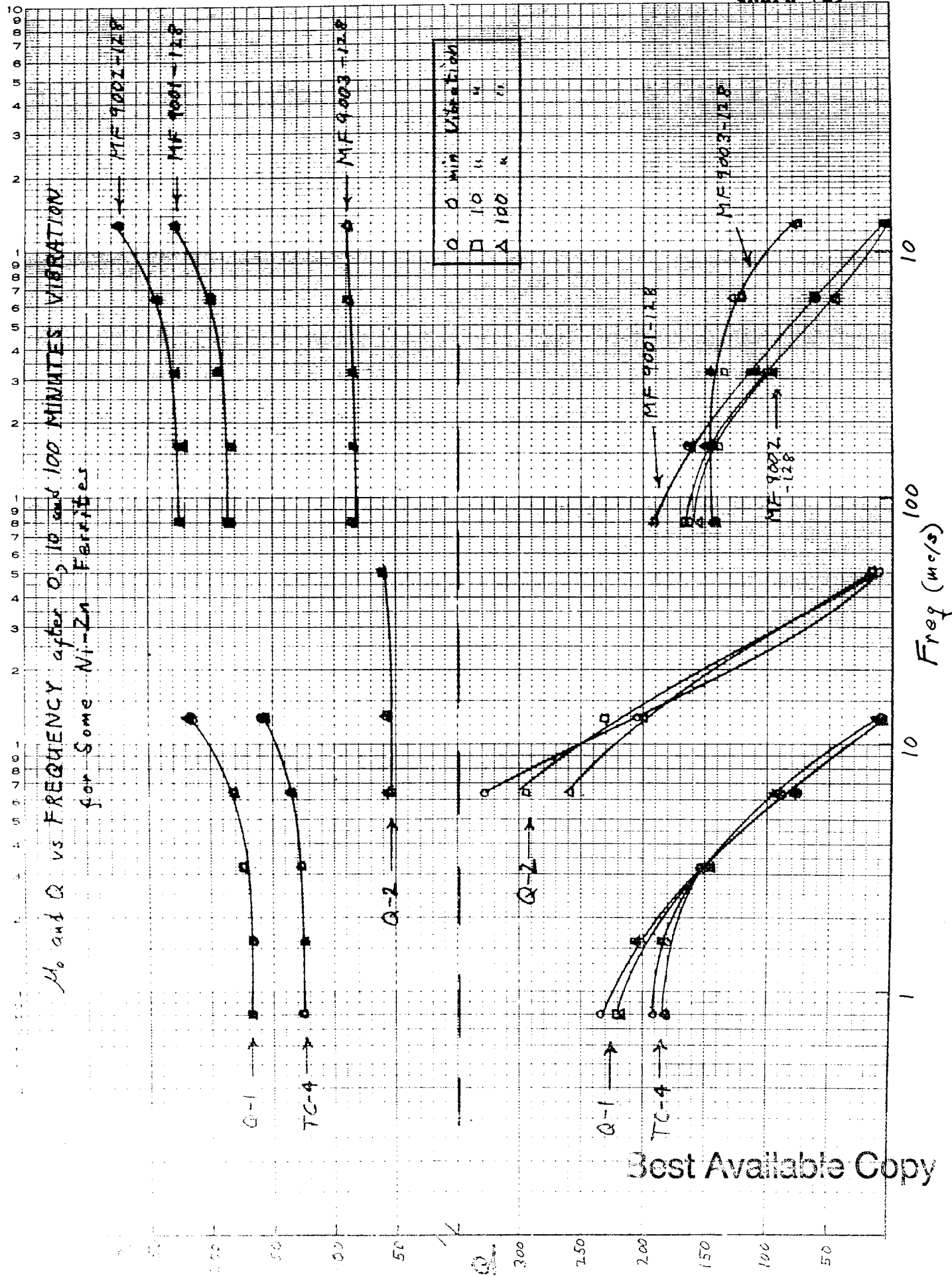
$\Delta \frac{\mu}{\mu_0}$ vs. TEMPERATURE FOR MATERIAL MP-8644-4
after 0, 10 and 100 MINUTES VIBRATION



Graph 712



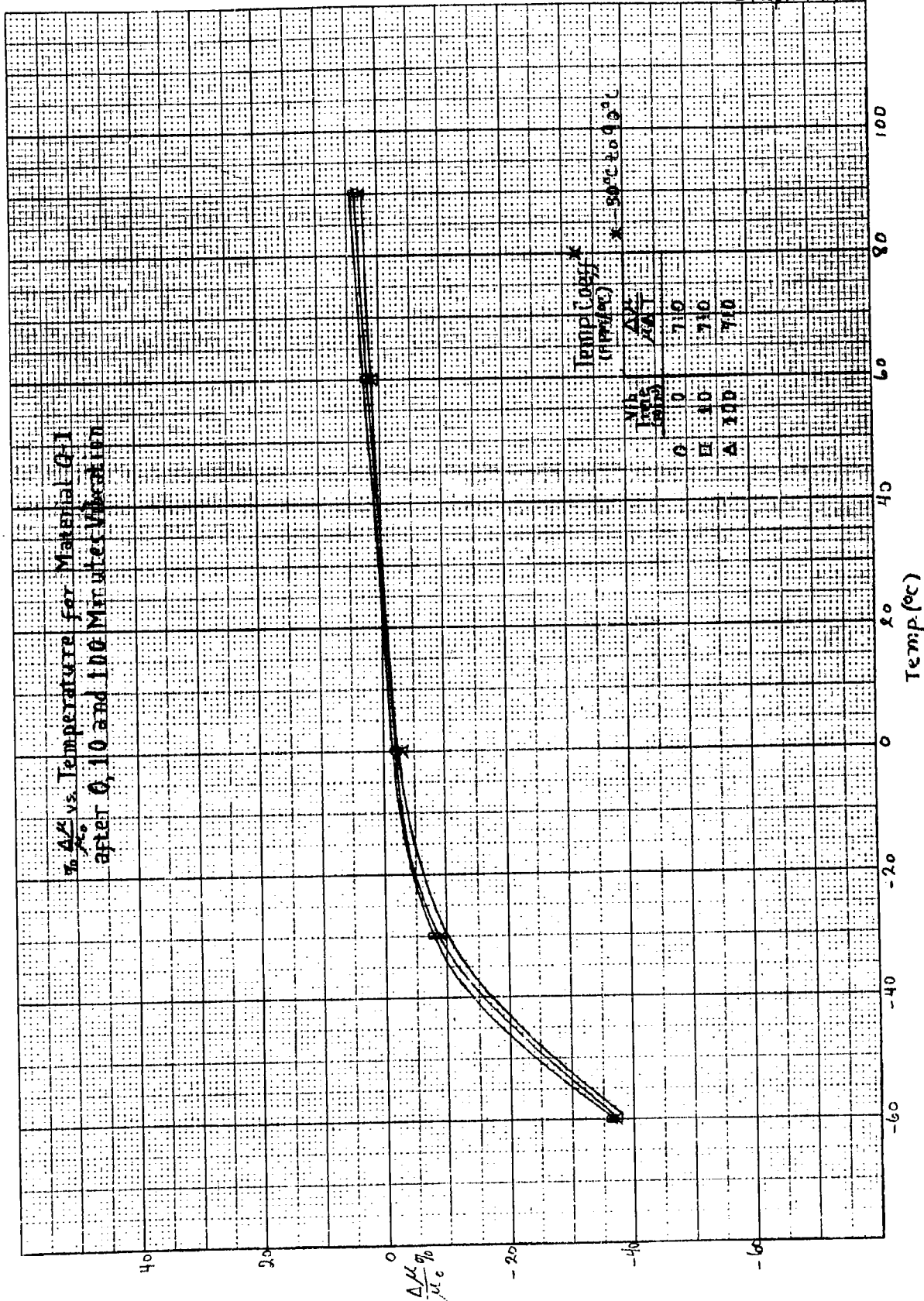
μ_0 and Q vs FREQUENCY after 0, 10 and 100 MINUTES VIBRATION
for Some Ni-Zn Ferrites



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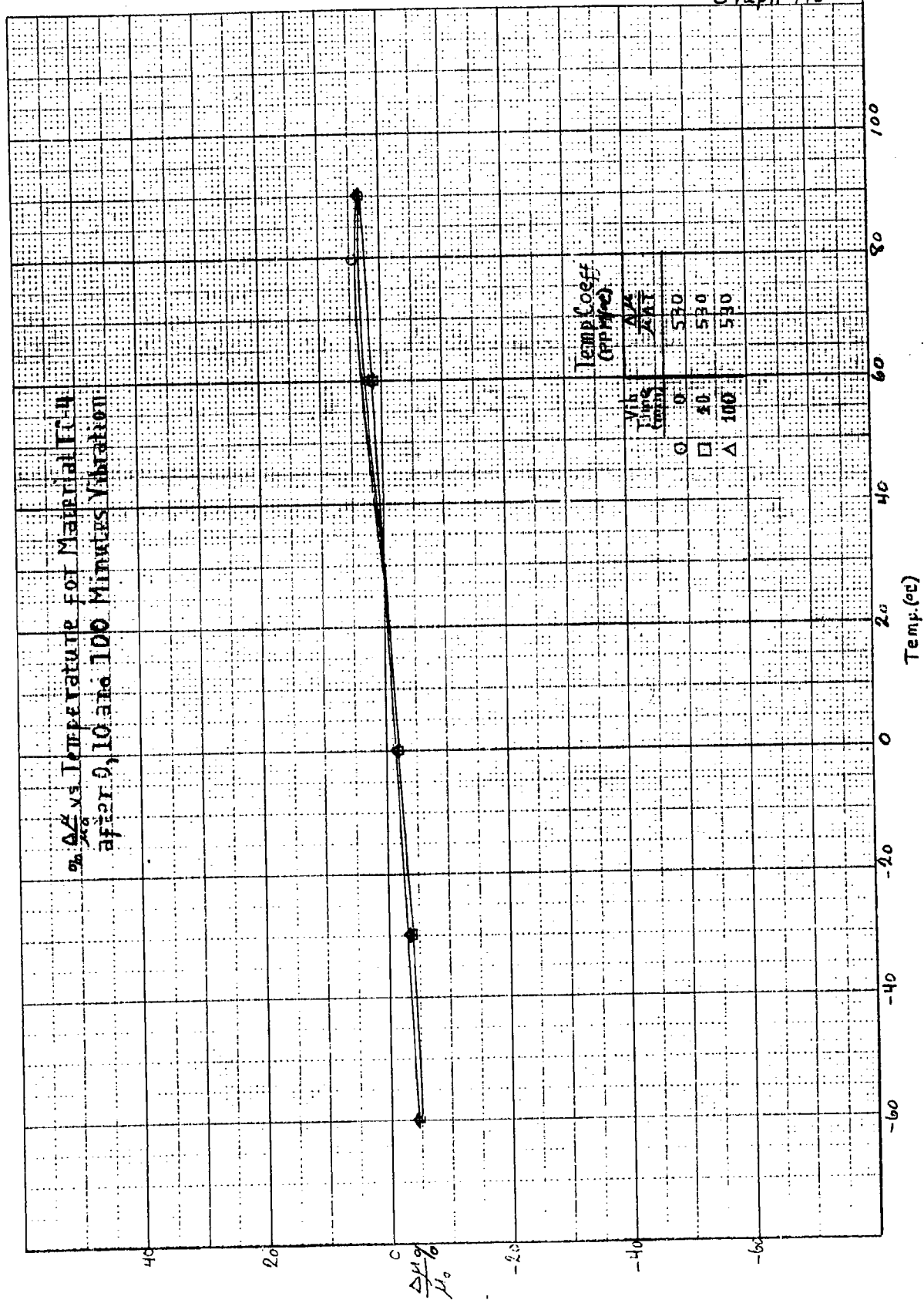
Graph 714

$\eta_0 \frac{\Delta \mu}{\mu_0}$ vs. Temperature for Material Q-1
after 0, 10 and 100 Minutes Vibration



Graph 715

% $\frac{\Delta \mu}{\mu_0}$ vs Temperature for Material F-4
after 0, 10 and 100 Minutes Vibration



Temp Coeff
(ppm/°C)

Vib. Time (min)

Δμ/ΔT

□

Δ

0

40

100

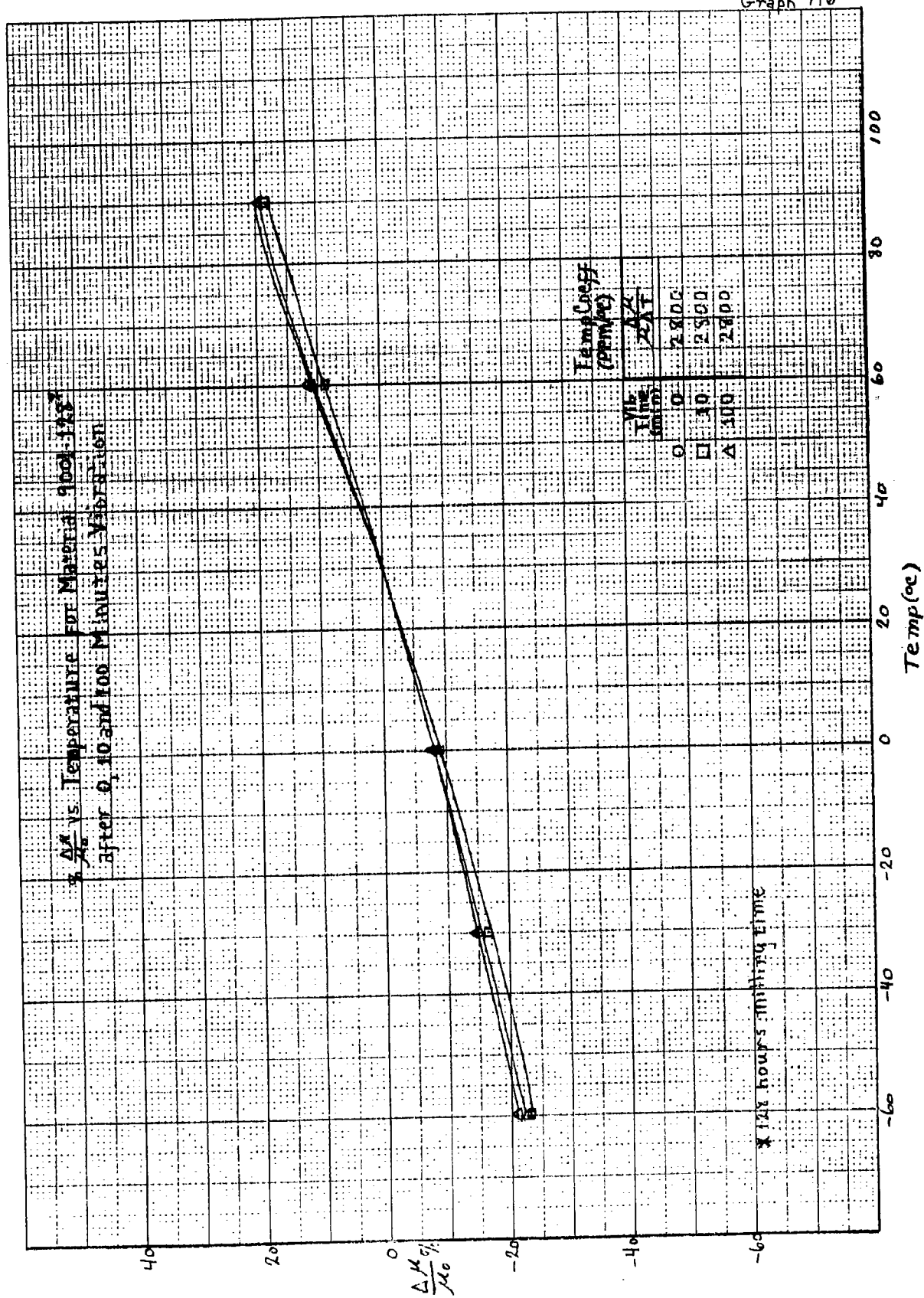
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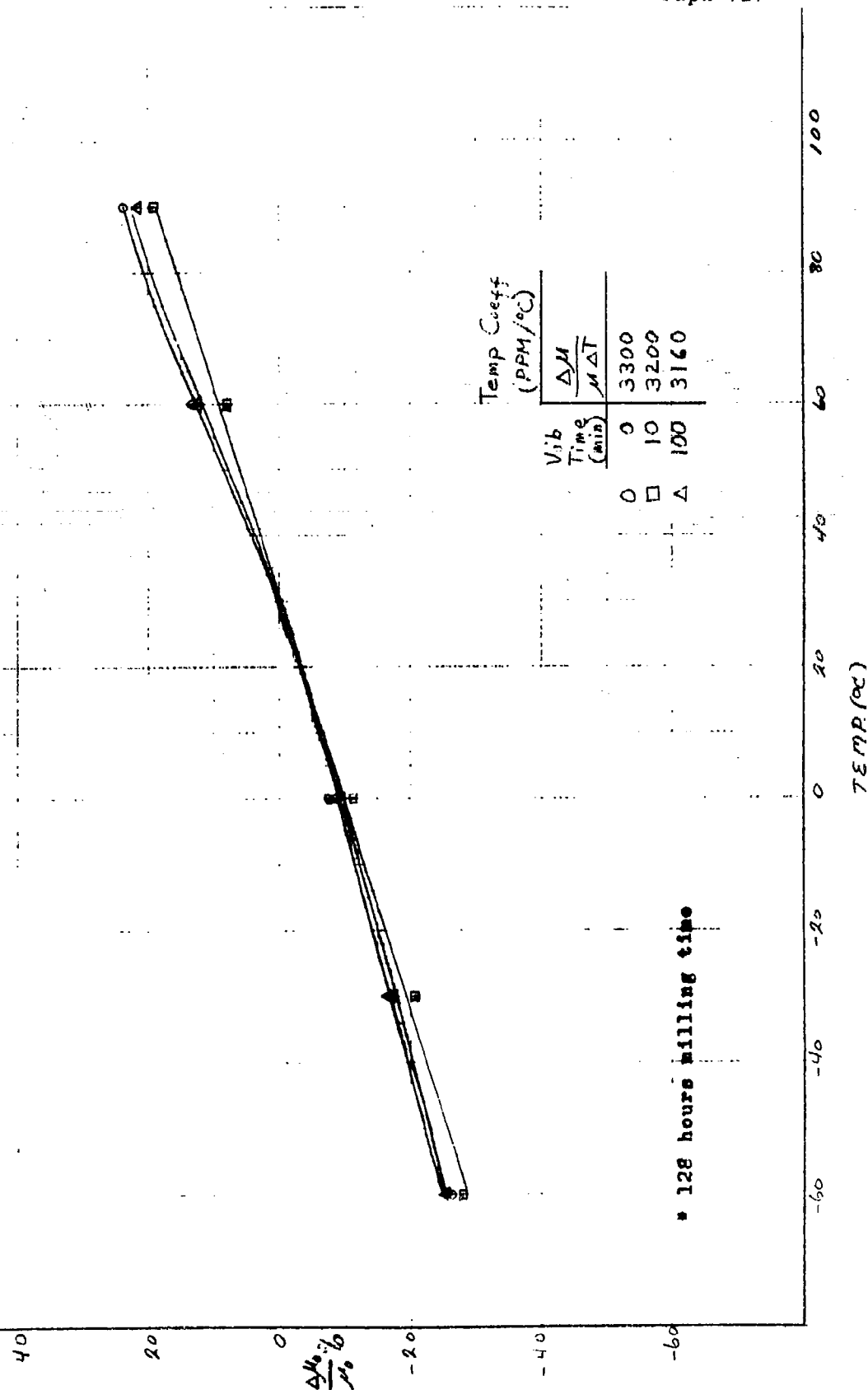
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Temp (°C)

Graph 716

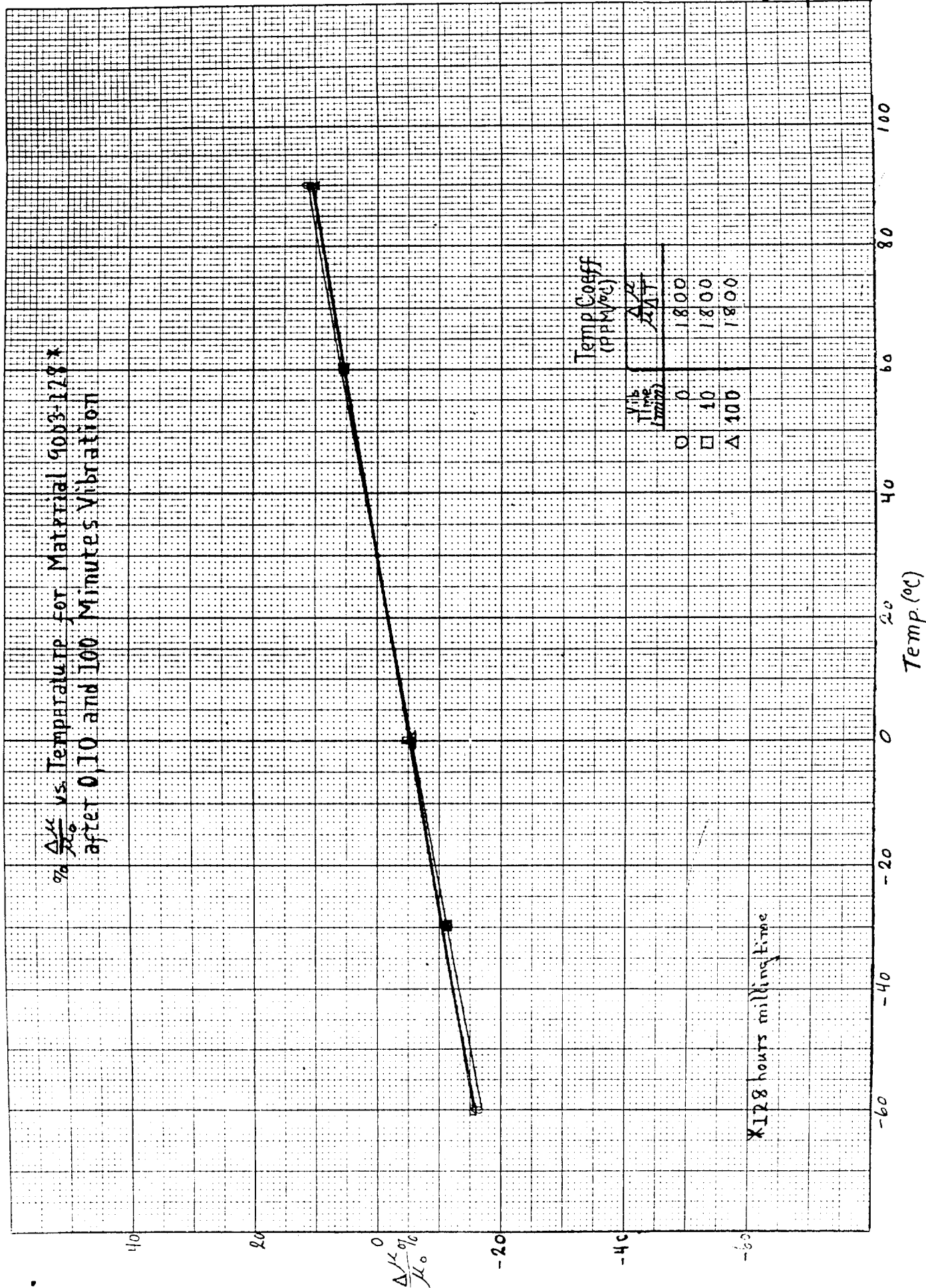


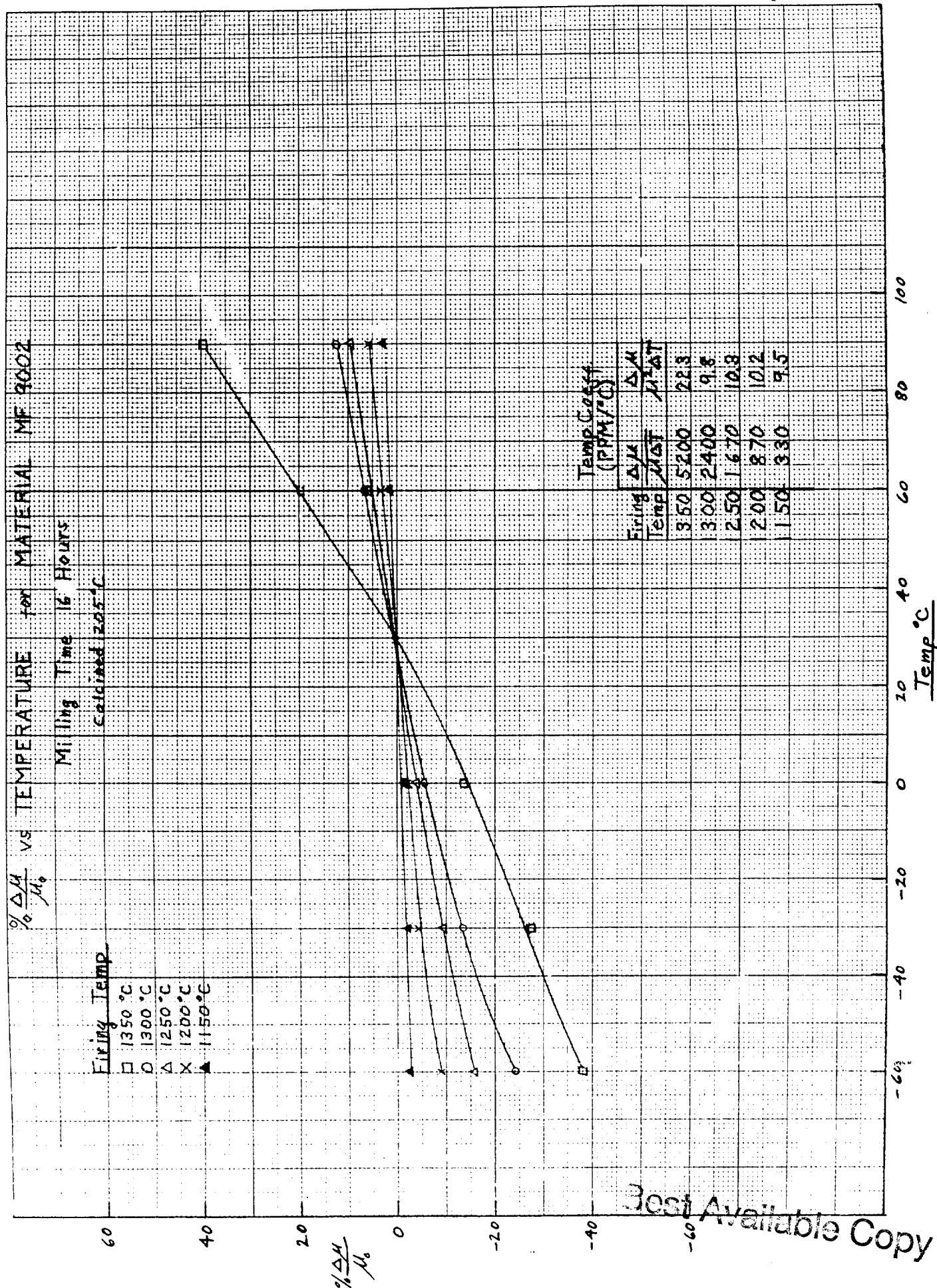
$\Delta \mu_0^0$ vs. TEMPERATURE for MATERIAL 9002-128*
after 0, 10 and 100 MINUTES VIBRATION

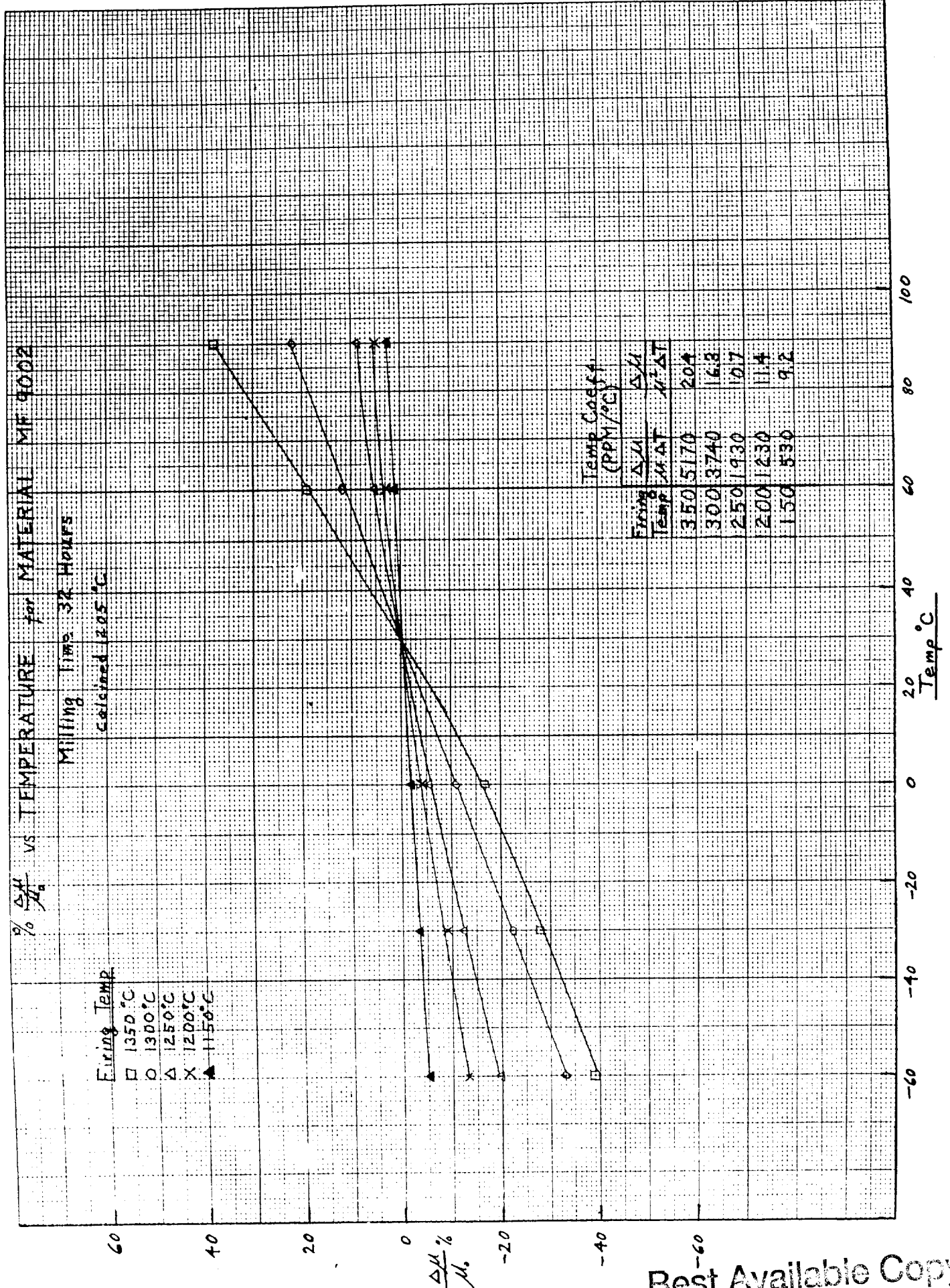


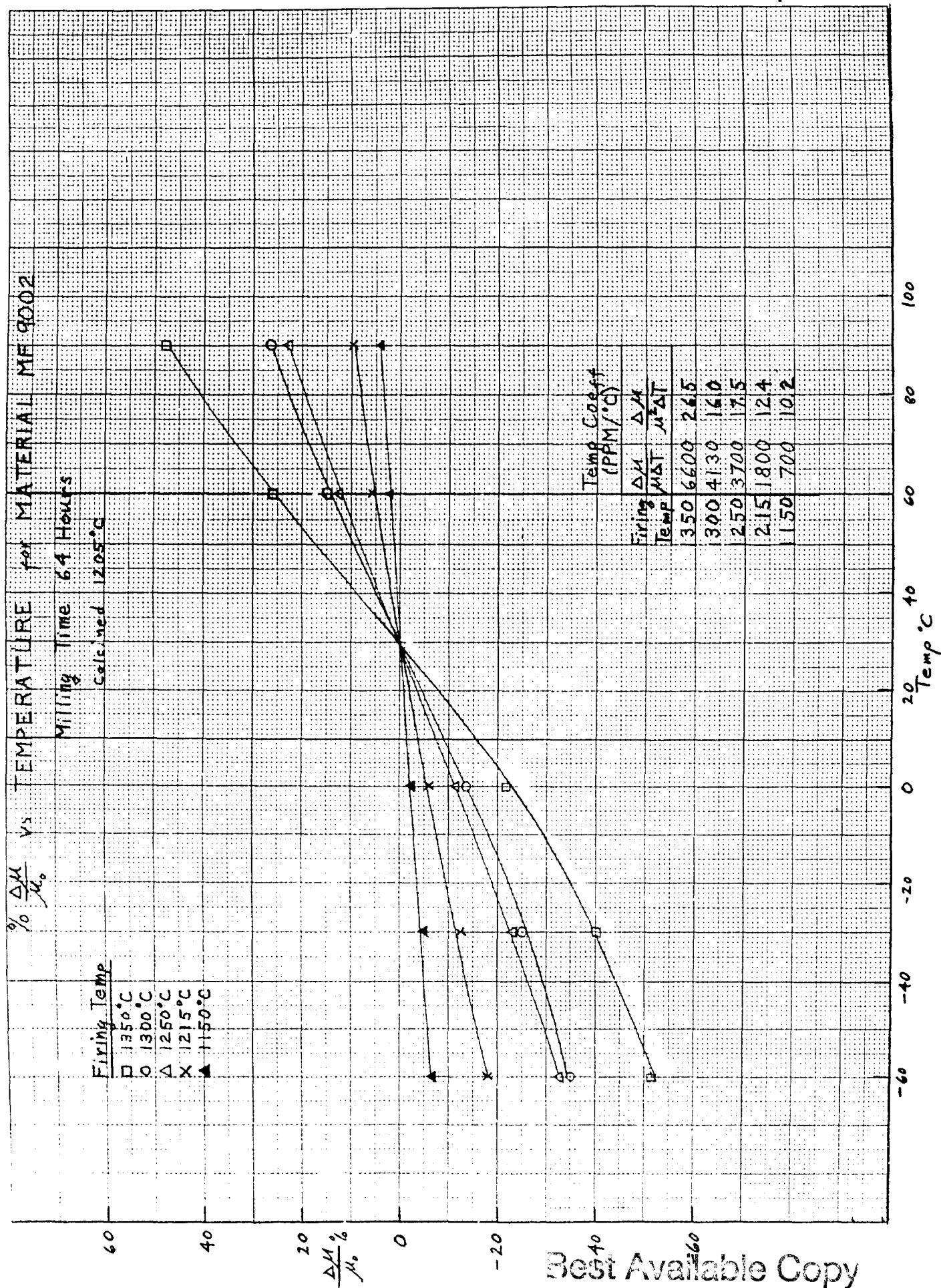
* 128 hours milling time

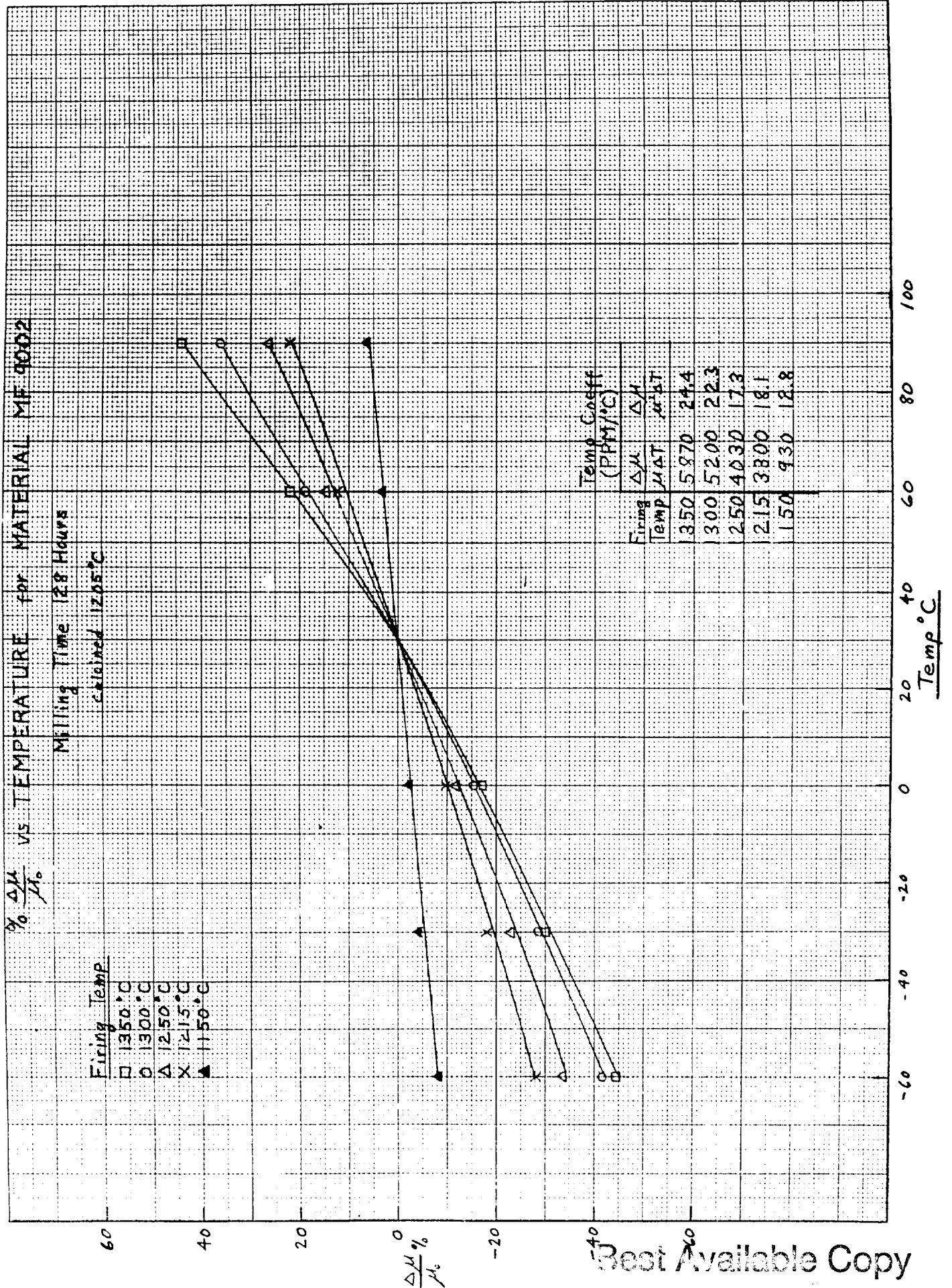
% $\frac{\Delta \mu}{\mu_0}$ vs. Temperature for Material 9003-123*
after 0, 10 and 100 Minutes Vibration

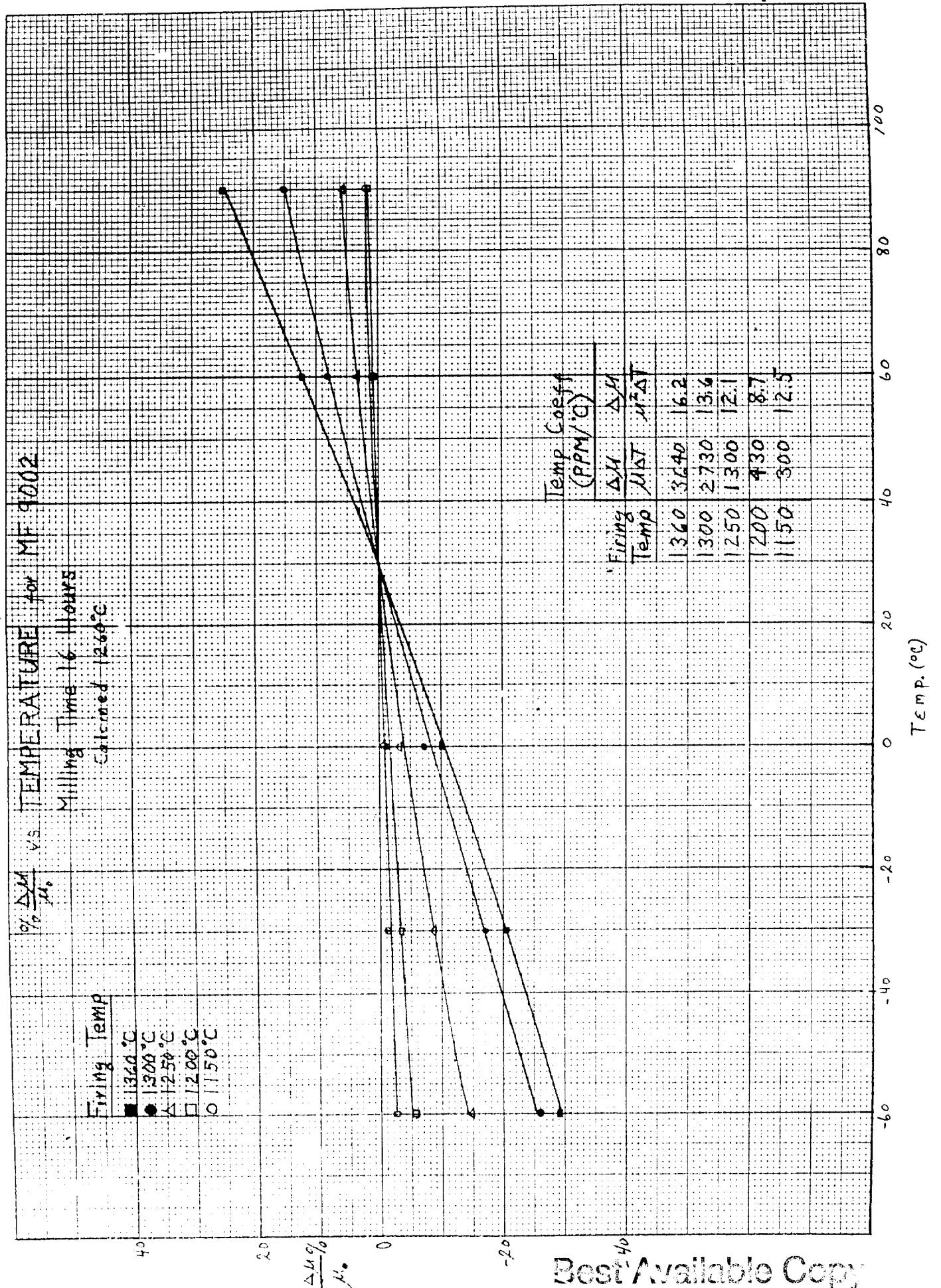




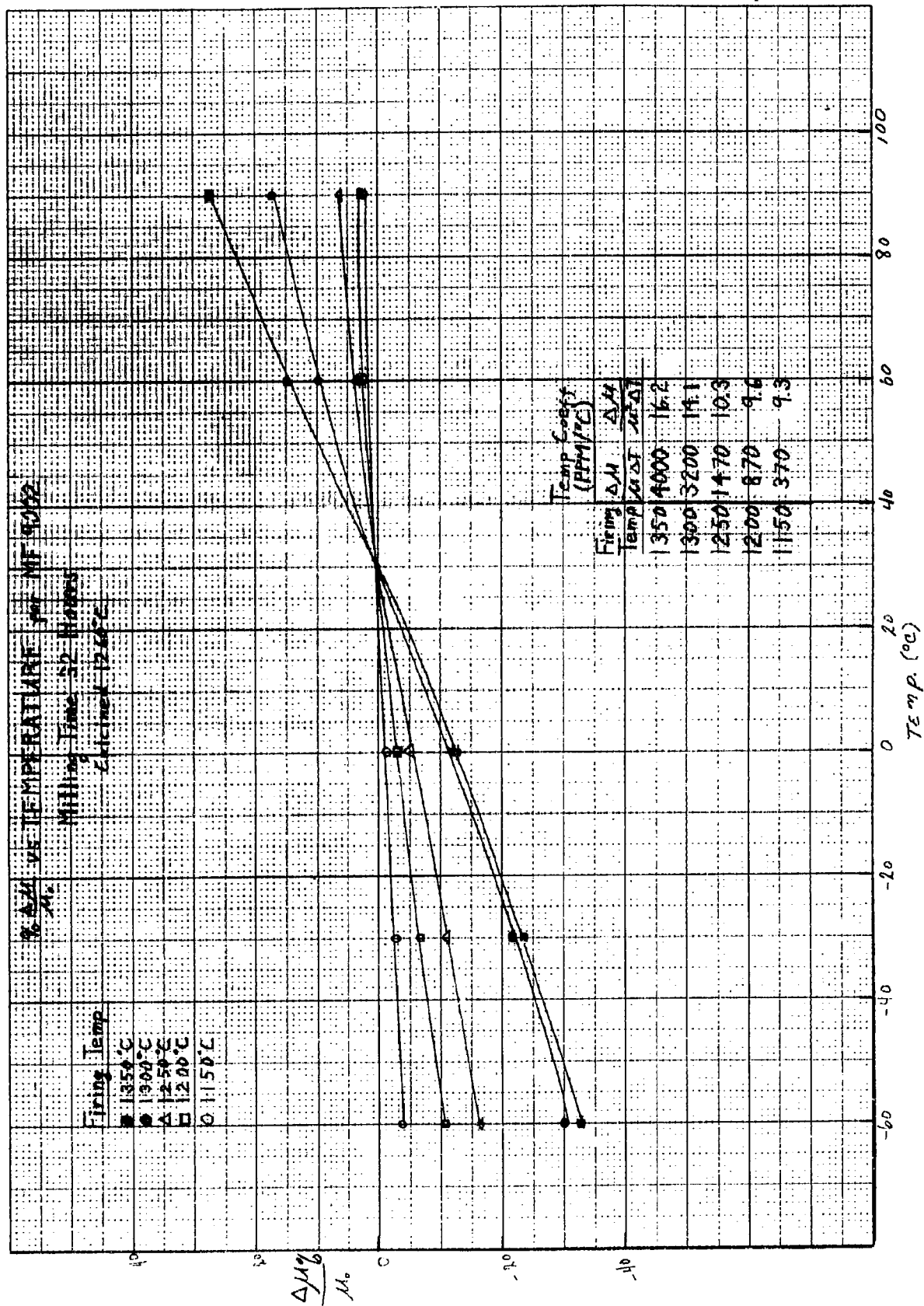


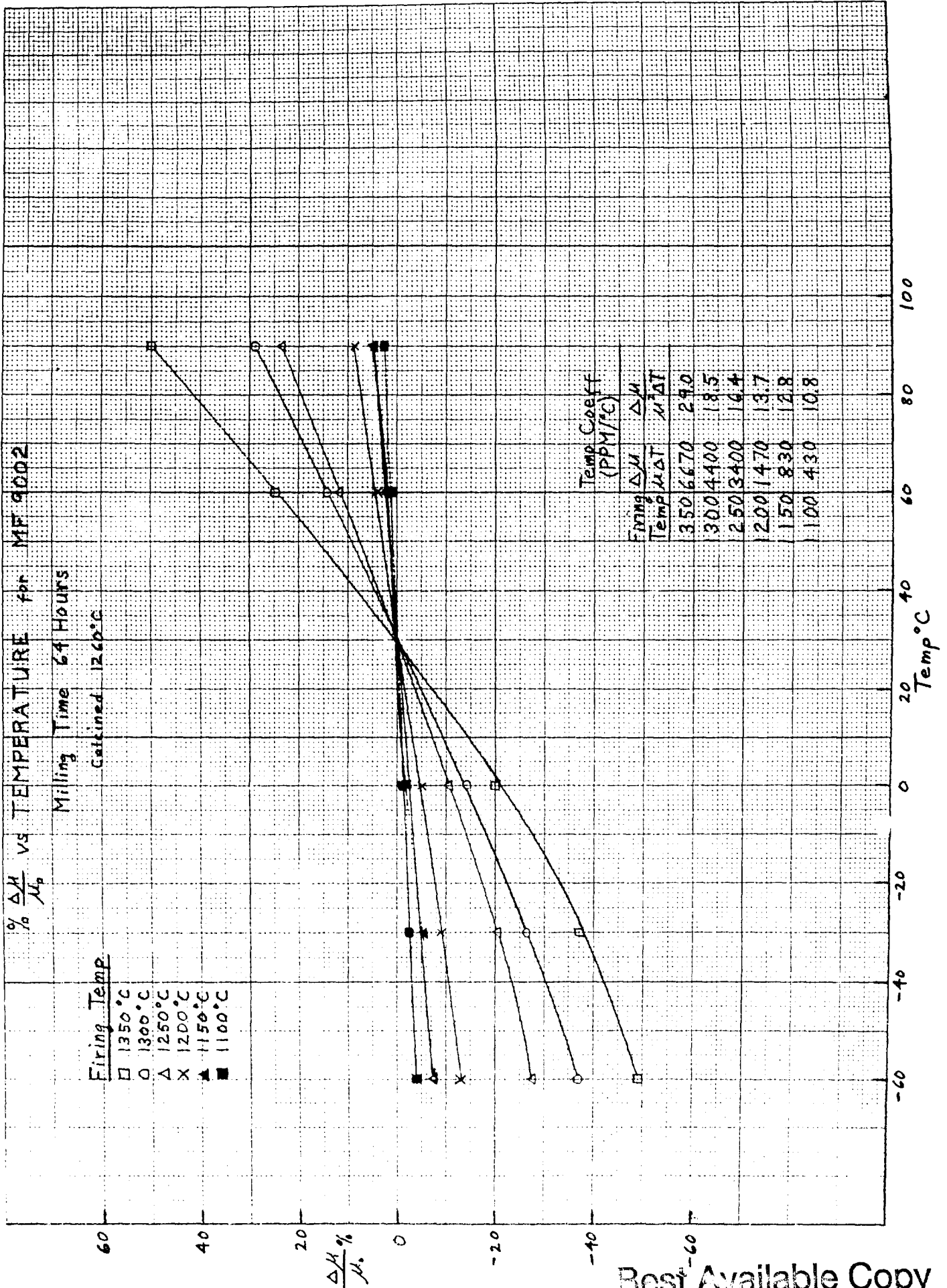


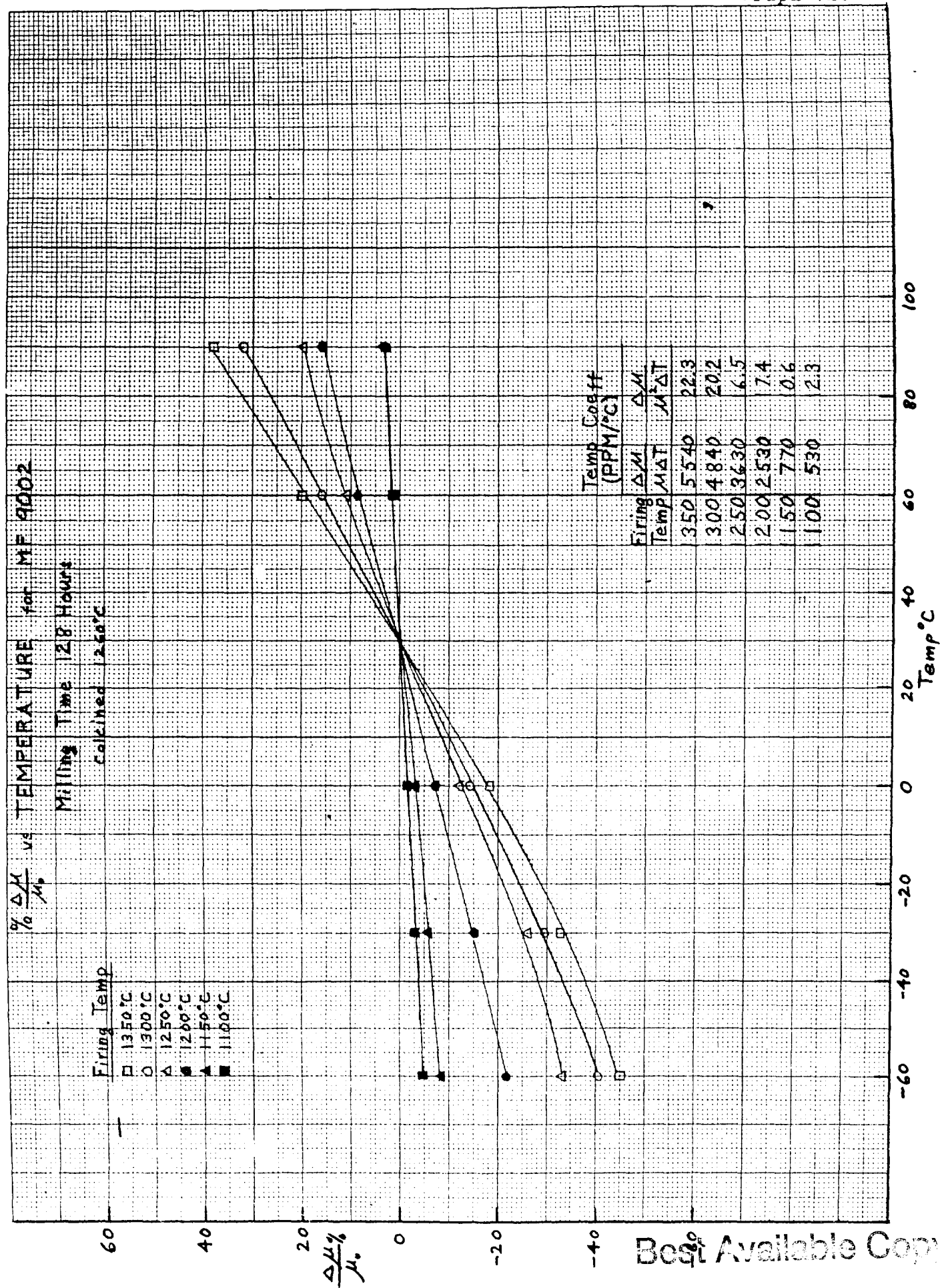




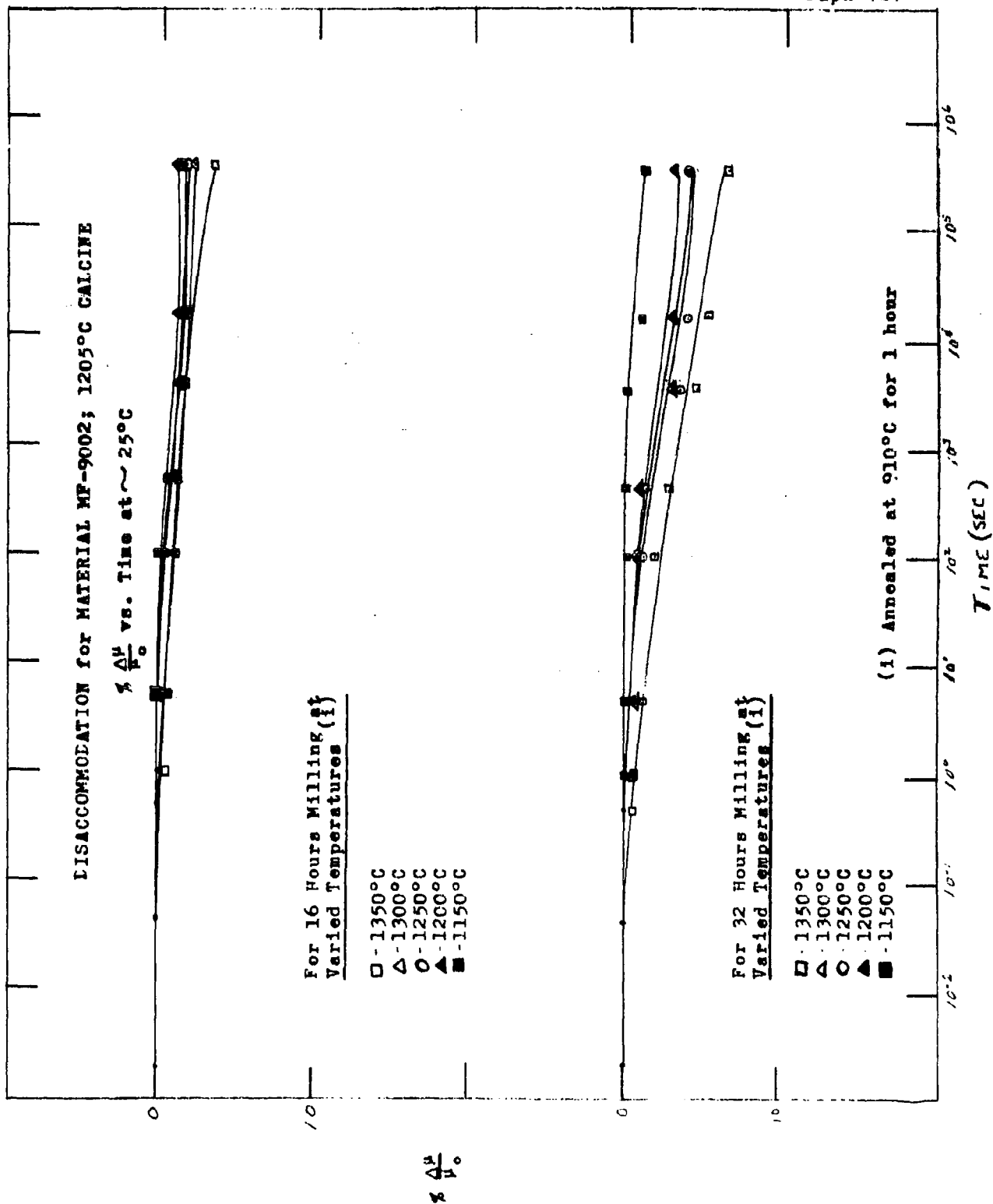
Graph 724

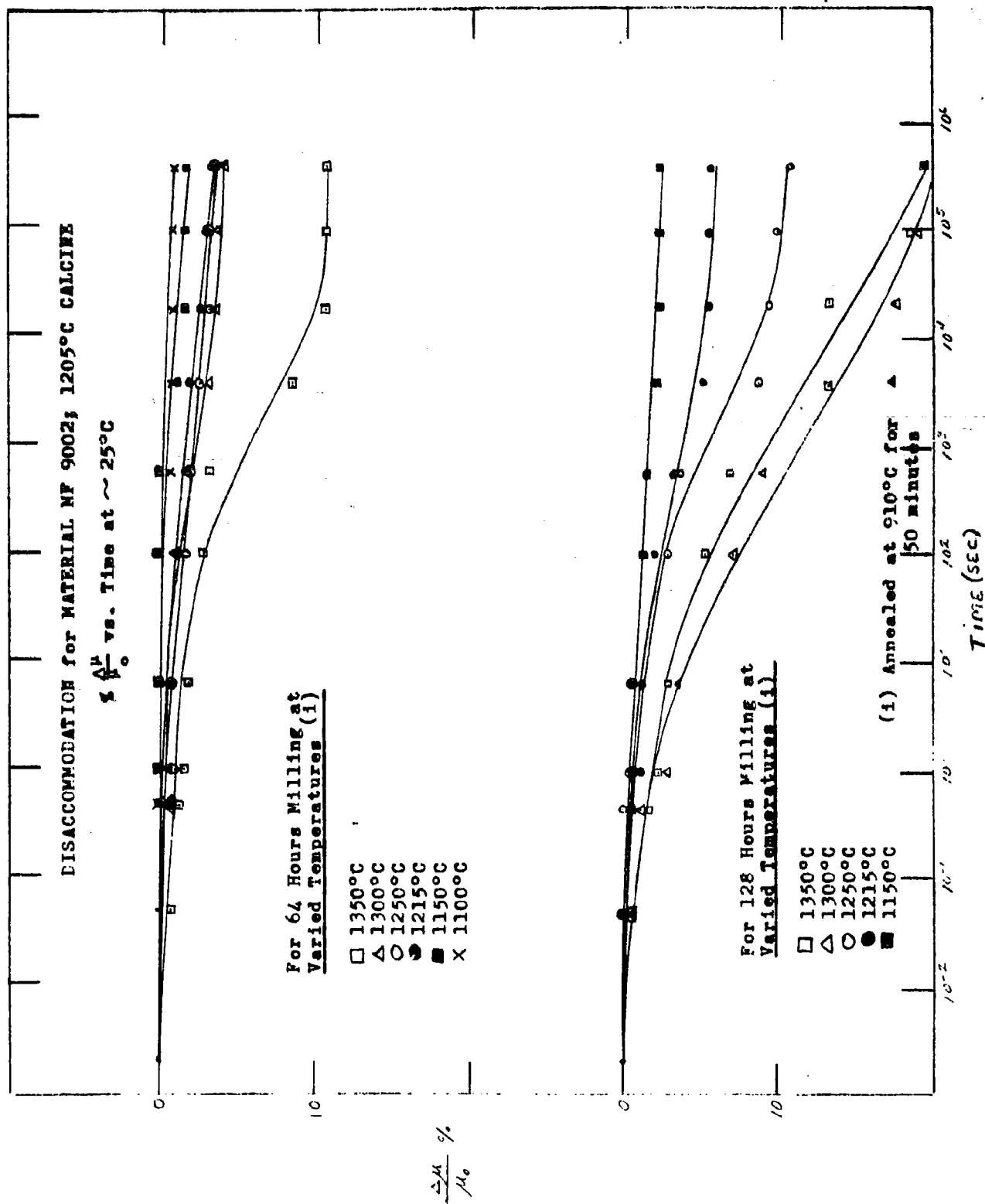


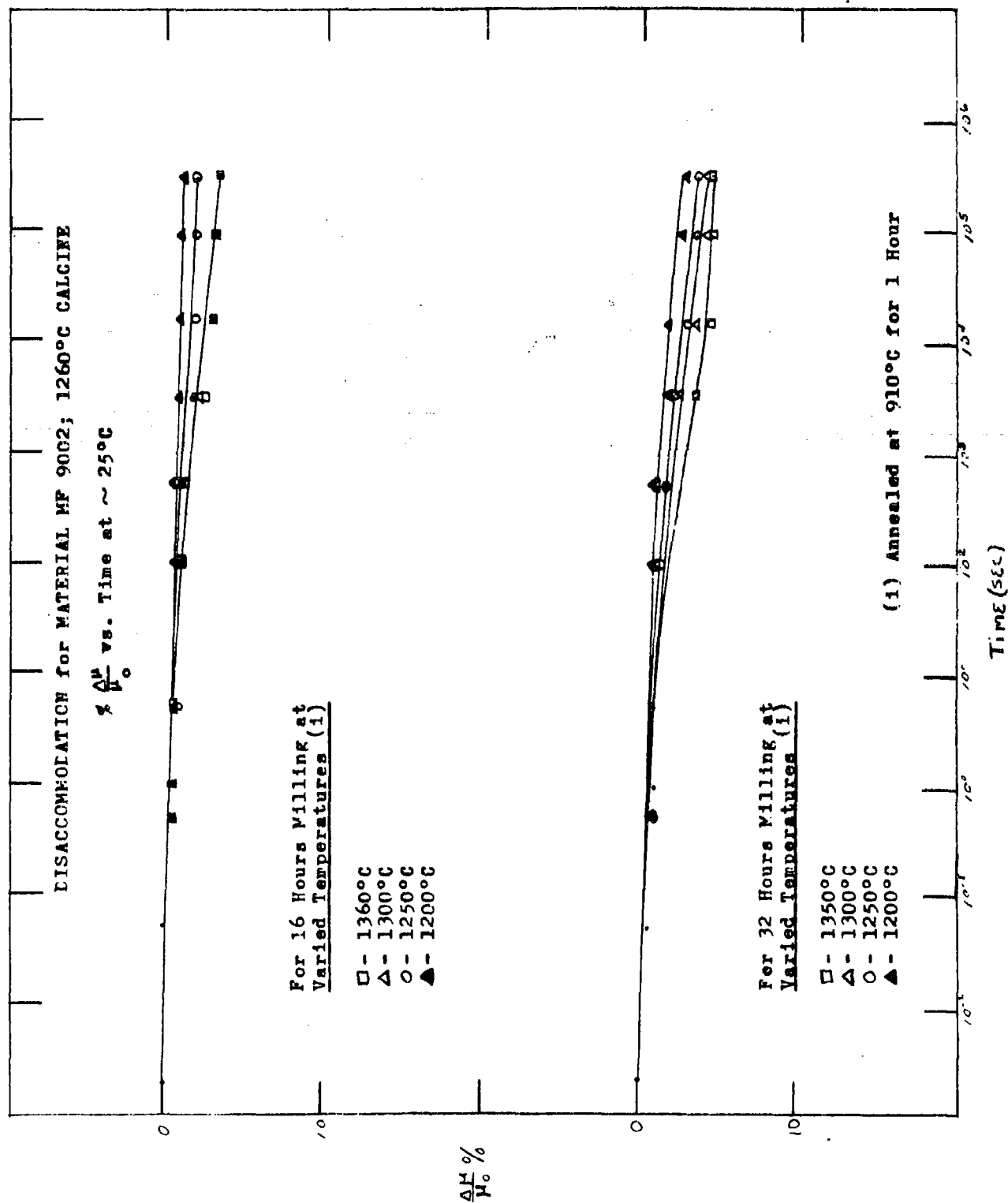




Graph 727







DISACCOMPOLATION FOR MATERIAL MF 9002; 1260°C CALCINE

 $\% \Delta H$ vs. Time at $\sim 25^\circ\text{C}$ Per 64 Hours Milling at
Varied Temperatures (i)

- 1350°C
- 1300°C
- 1250°C
- 1215°C
- 1150°C
- 1100°C

For 128 Hours Milling at
Varied Temperatures

- - 1350°C
- △ - 1300°C
- - 1250°C
- - 1200°C
- - 1150°C
- × - 1100°C

(i) Annealed at 910°C for 50 minutes

Time (sec)

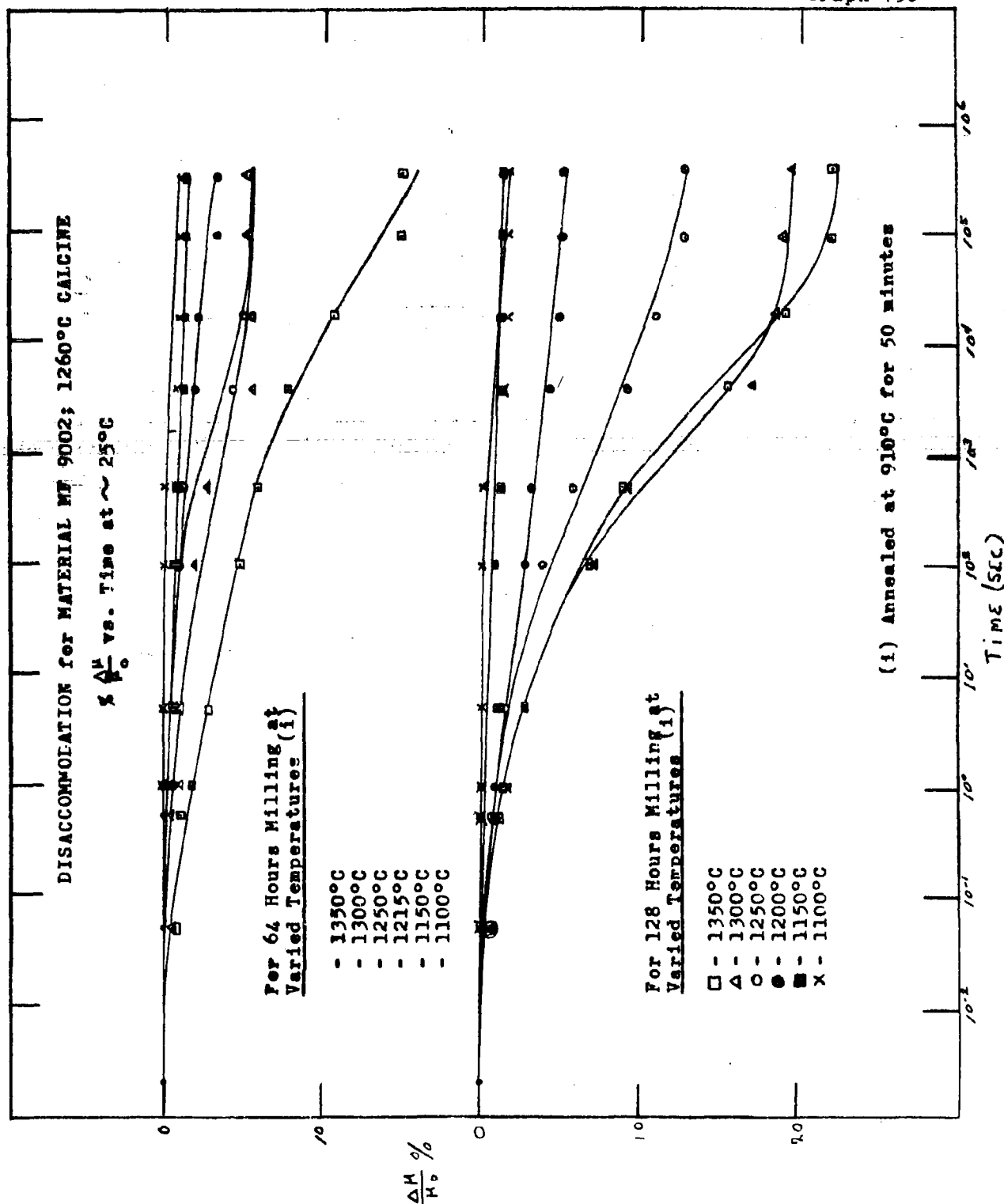


TABLE 296

THE PERMEABILITY CHANGE OBSERVED
AFTER 24 HOURS AT EACH OF THREE TEMPERATURES

Material	-40°C				25°C				50°C			
	Initial		After 24 hrs.		Initial		After 24 hrs.		Initial		After 24 hrs.	
	μ_0		%		μ_0		%		μ_0		%	
TC-5	1245	1218	2.2		1692	1669	1.4		1788	1784	0.2	
MF-8433-2	815	804	1.3		1129	1074	4.9		1150	1105	3.9	
MF-8644-4	824	805	2.3		1304	1260	3.4		1405	1376	2.1	
Q-1	137	136	0.7		184	182	1.1		187	179	4.3	
Q-1 (not demagnetized)	123	123	0.0		146	146	0.0		149	148	0.7	
TC-4	127	127	0.0		142	140	1.4		146	139	4.8	
TC-4 (not demagnetized)	104	104	0.0		108	108	0.0		110	110	0.0	
MF-9001-128	142	141	0.7		172	170	1.2		186	185	0.5	
MF-9002-128	73	72	1.4		90	89	1.1		99	99	0.0	

TABLE 297

PHYSICAL AND MAGNETIC DATA FOR MATERIAL MP-9002 FOR VARIOUS CONDITIONS OF MILLING AND FIRING
(1205°C Calcine)

Mol Formula (%)														
Fe ₂ O ₃ 50.00														
ZnO 25.00														
PbO 25.00														
MILLING TIME (HOURS)	FIRING TEMP. (°C)	CRYSTAL SIZE (μ)	DENSITY (g/cc)	(6.4 mc/s)			UPPER USEFUL FREQ. (mc/s)	μEQ. UNF. 10 ⁻³	REF. 11 GRAPH	PA(%) ¹¹¹ CYCLE	PA(DPM) CYCLE-μO	Temp. Coef. (ppm/°C)		
				μO	Q	μEQ. 10 ⁻³						Δμ ΔT	μΔT	
16	1150		3.80	37	59.6	2.19	14.3	13.9		.4	115.0	330	9.5	
	1200		4.06	95	44.4	4.20	7.6	24.9		.5	60.9	870	10.2	
	1250		4.47	187	20.9	3.91	4.4	32.3	297	.5	32.1	1670	10.3	
	1300	2.4	4.77	297	9.0	2.68	3.5	38.6		.4	16.3	2400	9.8	
	1350		4.86	266	5.2	1.38	2.8	15.9		1.0	42.9	5200	22.3	
32	1150		4.09	61	79.7	4.85	15.8	32.7		.5	83.6	530	9.2	
	1200		4.31	119	67.5	8.03	9.9	47.2		.9	81.5	1230	11.4	
	1250		4.67	209	33.9	7.09	4.9	54.0	297	1.0	57.4	1930	10.7	
	1300	2.0	4.93	268	16.4	4.40	3.9	47.2		1.1	46.9	3740	16.3	
	1350		4.91	301	5.4	1.63	2.9	22.9		1.1	42.5	5170	20.4	
64	1100		3.88	47	68.1	3.21	21.0	2.9		.3	72.7			
	1150		4.09	73	77.8	5.66	14.8	37.4		.5	76.3	700	10.2	
	1215		4.58	160	66.1	10.60	8.0	65.5		.7	46.9	1800	12.4	
	1250	1.9	4.90	251	24.9	6.25	4.4	55.9	298	.6	26.4	3700	17.5	
	1300		4.93	312	11.9	3.71	3.5	47.6		.9	35.6	4130	16.0	
1350		4.90	278	3.8	1.06	2.9	14.7		3.2	128.5	6600	26.5		
128	1100			54	83.6	4.50	23.8	44.5						
	1150		4.56	77	89.3	7.61	16.2	53.0		.3	44.3	930	12.8	
	1215	.9	4.79	202	66.0	13.30	6.9	83.5	298	1.3	72.5	3300	18.1	
	1250		4.86	271	16.3	4.42	3.6	50.2		3.1	132.2	4030	17.3	
	1300		4.86	266	5.9	1.56	2.8	23.1		4.2	180.3	5200	22.3	
1350		4.84	273	4.9	1.32	2.8	18.8		4.1	170.2	5870	24.4		

i. Annealed 910°C, 1 hour
ii. Report No. 56
iii. DA - Disaccommodation

TABLE 298

CHEMICAL ANALYSIS OF MATERIAL MF-9002
AFTER CALCINING AND MILLING

Starting Formula Wt. % Mol %

Fe ₂ O ₃	67.42	50.30
ZnO	17.25	25.25
NiO	15.33	24.45

1205°C Calcine

	<u>Composition in wt. %</u>				<u>Composition in mol %</u>			
	<u>Milling Time (hrs.)</u>				<u>Milling Time (hrs.)</u>			
	16	32	64	128	16	32	64	128
Fe ₂ O ₃	68.19	69.23	69.75	71.20	51.21	52.42	53.02	54.77
ZnO	17.17	16.73	16.37	15.61	25.30	24.86	24.43	23.55
NiO	14.64	14.04	13.88	13.19	23.49	22.72	22.55	21.68

1260°C Calcine

	<u>Composition in wt. %</u>				<u>Composition in mol %</u>			
	<u>Milling Time (hrs.)</u>				<u>Milling Time (hrs.)</u>			
	16	32	64	128	16	32	64	128
Fe ₂ O ₃	67.64	68.85	70.02	70.96	50.55	51.94	53.56	54.45
ZnO	17.06	16.51	15.89	15.54	25.00	24.44	23.40	23.40
NiO	15.30	14.64	14.09	13.50	24.45	23.62	23.04	22.15

TABLE 299

CHEMICAL ANALYSIS OF MATERIAL MF-9001-A
AFTER CALCINING AND MILLING

<u>Starting Formula</u>	<u>Wt. %</u>	<u>Mol %</u>
Fe ₂ O ₃	66.89	49.88
ZnO	20.14	29.46
NiO	12.95	20.64

1205°C Calcine

	<u>Composition in wt. %</u>		<u>Composition in mol %</u>	
	<u>Milling Time (hrs.)</u>		<u>Milling Time (hrs.)</u>	
	<u>64</u>	<u>128</u>	<u>64</u>	<u>128</u>
Fe ₂ O ₃	69.72	71.20	51.86	54.88
ZnO	18.26	17.25	29.03	26.10
NiO	12.02	11.54	19.11	19.02

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